



The Significant Role of Mxene's Derivatives and Composites in Supercapacitors: A Review

Aknur S. Berkinbayeva,¹ Meruyert Nazhipkyzy,^{2,3,4,*} Muhammad Hashami,^{2,3,5} Zhanar B. Kudyarova,^{2,6} Togzhan Mashan,⁷ Aigul S. Beisebayeva,¹ Kenes K. Kudaibergenov¹ and Madina Auyelkhankyzy²

Abstract

MXene derivatives and composites are becoming fresh and promising two-dimensional (2D) materials because of their unique properties, performance, and multifunctionalities. This review covers the general aspects of MXenes (family of 2D transition metal carbides, nitrides, and carbonitrides), their MAX phase (a diverse class of nanolaminate materials with intriguing properties that have received incredible global research attention because they bridge the divide separating metals and ceramics.) precursors, dissimilarities and similarities between MAX phases and MXenes, and special properties of both materials. The important focuses are made on the methods of MXene synthesis and their stability and conductivity, as well as on synthesizing new MXene composites with desired characteristics. The impacts of MXenes in energy storage systems, especially supercapacitors, are discussed based on the high specific surface area, tunable porosity, and optimized inter-layer spacing. Here, various aspects of MXene materials, including properties, synthesis, and applications, have been discussed to highlight their importance for the development of energy storage systems and material science research.

Keywords: MAX phase; Mxene; MXene composites; Supercapacitors.

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1. Introduction

Nanostructured materials have attracted the interest of scientists in recent years due to their novel characteristics at the nanoscale. According to studies, these features alter depending on their form and size. These particles are generally defined as solid materials with at least one dimension in the nanoscale range. The dimension of nanostructures is a major feature that separates nanostructures into several categories, as well as an important aspect of the material that determines the atomic structure and establishes properties to a significant

extent. Nanomaterials can be divided into four categories based on the dimension criteria: zero-dimensional (0D), one-dimensional (1D), two-dimensional (2D), and three-dimensional (3D). The types, structures, and examples of nanomaterials are shown in Fig. 1. 0D nanomaterials are materials having all dimensions within the nanoscale, 1D nanomaterials are materials having one dimension outside the nanoscale, 2D nanomaterials are materials having two dimensions outside the nanoscale, and 3D materials are materials that don't confine to the nanoscale in any dimension. Nowadays, 2D nanomaterials are deeply studied due to their unique properties, such as electronic and optical properties, and increasing innovative applications in supercapacitors, energy storage, photocatalysts, sensors, nanoreactors, and nanocontainers.^[1]

As depicted in Figs. 1 and 2, 2D nanomaterials share similar structures, but their unique characteristics have successfully piqued the interest of researchers worldwide. Their vast potential has driven the development of an extensive field dedicated to 2D materials. These materials have a high atomic-scale thickness, which results in a high surface-to-volume atomic surface density. Unlike graphene, a semimetal with a zero or near-zero bandgap, 2D materials are typically semiconductive.^[2]

¹ Satbayev University, Satbayev Str. 22, Almaty, 050000, Kazakhstan

² Al-Farabi Kazakh National University, al-Farabi Ave 71, Almaty, 050040, Kazakhstan

³ Institute of Combustion Problem, Bogenbay Batyr Str. 178, Almaty, 050012, Kazakhstan

⁴ Imperial College London, London, SW7 2AZ, United Kingdom

⁵ Institute of Higher Education Mirwais Khan Nika Zabul, Qalat, 041325, Afghanistan

⁶ Kazakh National Women's Teacher Training University, Gogol Str. 114, Almaty, 050000, Kazakhstan

⁷ L.N. Gumilyov Eurasian National University, Kazhymukan Str. 11, Astana, 010000, Kazakhstan

*Email: meruert82@mail.ru (M. Nazhipkyzy)

This manuscript delves deeply into MXenes, summarizing the latest computational and experimental studies and reflecting the rapid advancements within the MXene research community. MXenes have the general formula $(M_{n+1}X_nT_x)$, where M represents a transition metal (such as Ti, V, Ta, Zr, Hf, Cr, Mo, *etc.*), X refers to C, N, or both, and T_x represents functional groups such as hydroxyl, oxygen, or fluorine on the surface. MXenes are prepared by carefully etching the A element from a MAX phase, a form of ternary ceramic.^[3] MXenes were first reported in 2011 and have since been explored in various fields, including electronics,^[4] environmental science,^[5] and energy storage.^[6] These highly conductive and capacitive materials have a large surface area, making them suitable for these applications.^[7]

In energy storage, MXenes have been studied as electrode materials for devices such as supercapacitors and lithium-ion batteries. Unlike conventional electrode materials, MXenes offer advantages in electrical conductivity, charge transfer (enabling quick charge and discharge cycles), and a high surface area (which allows for greater charge accommodation in the interlayer space). Additionally, their flexibility and low density make them ideal for wearable and portable electronic devices.^[6] MXene-derived materials are composites that vary in terms of elemental terminations, structures, or doping. MXene composites are created by dispersing MXenes with other materials to improve efficiency in specific applications.^[8]

Despite extensive research, challenges remain in optimizing the performance and scalability of MXenes for practical applications. MXenes are prone to degradation under ambient conditions, limiting their long-term stability. Current synthesis methods, especially HF etching, face scalability, and environmental safety challenges. Furthermore, the limited voltage window (0.6–1.2 V in aqueous systems) restricts their energy density.

This review discusses the properties and potential of MXene derivatives and composites, with a particular focus on their application in supercapacitors. The aim is to analyze the current state of research, providing a comprehensive understanding of synthesis, characteristics, recent findings, and prospects for MXenes in practical applications, especially in supercapacitors. This work is intended to assist young researchers in identifying possible solutions to the challenges currently faced in the field.

2. Materials

2.1 MAX phase

MAX phases are layered hexagonal-shaped atom arrangements of materials, in the structure of MAX phases where M is a transition metal, A is a group 13-16 (Al, Cd, P, Si, Ge, Ga, In, As, Tl, Sn, Pb, S), and X is either carbon or nitrogen. The general formula for the structure is $M_{n+1}AX_nT_x$, where T is a surface termination group, and n is the number of layers of edge-sharing. The close-packed plane of atom A is situated between the MX layers. Another way to think of the structure is as a stacking of n+1 atomic planes in the order

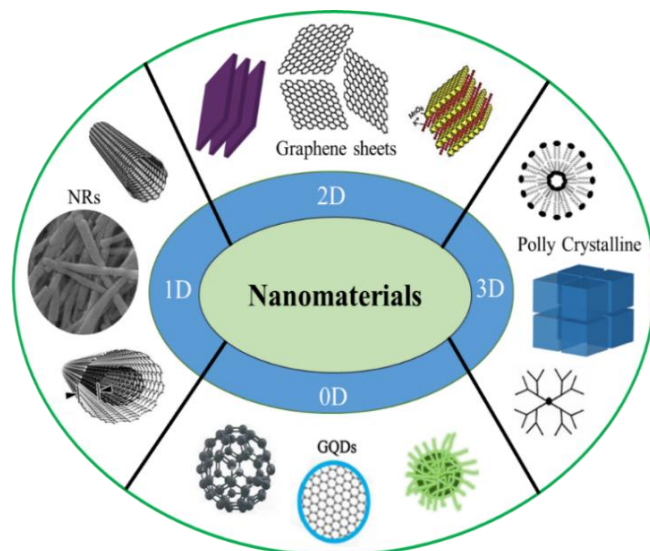


Fig. 1: 0D (fullerene, carbon dots, nanoparticles), 1D (single- and multi-walled nanotubes, nanofibers, nanowiskers, nanorods, nanobelts, nanowires), 2D-nanomaterials (MXene, graphene, carbon nitrides, metal-organic frameworks (MOFs)) and 3D materials (polycrystalline materials such as polycrystalline silicon, carbon aerogels and foams).

$(MX)_n(MA)_x$ along the c-axis. For instance, Ti_2AlC , a 211 MAX phase, can be characterized by high stiffness, hardness, thermal conductivity, electrical conductivity, and machinability. It can be employed as a structural component in high-temperature devices, including nuclear reactors, gas turbines, and aircraft engines.^[8] Ti_3SiC_2 is a 312 MAX phase characterized by minimal friction, self-crack healing, high toughness, and thermal shock resistance. It has multiple applications such as bond and protective coating for gas turbines, catalyst for carbon dioxide reduction and water splitting, and joining material for metals and ceramics. Ti_4AlN_3 is a 413 MAX phase characterized by strong oxidation resistance, damage tolerance, and radiation tolerance. It can be employed as an electrical interface for high-voltage applications such as solar receivers in concentrated solar systems and an accident-tolerant fuel cladding in nuclear power plants.^[9,10] Mo_4VAlC_4 is a 514 MAX phase characterized by strong electrical conductivity, hardness, and thermal stability. It has multiple applications, such as high-temperature electrode material for batteries and supercapacitors, wear-resistant material for cutting tools, and thermoelectric material for energy conversion.

2.2. MXene

MXene, a 2D nanomaterial that is exfoliated from layered MAX phases, has garnered increasing interest in recent years due to its physical and chemical properties. This growing interest is illustrated by the histogram shown in Fig. 2a. The advances achieved in preparation and properties, together with the explored applications of MXene, provide a strong impetus to further progress in the characterization and applications of these new 2D materials, as shown in pie-chart in Fig. 2b.

2.3 Characteristics of MXene

MXene possesses mechanical, thermal, chemical, electrical, and magnetic properties, which set it apart from innovative materials. It is known for being hard and having a high Young's modulus while also being a ductile material with low fracture toughness.^[11] Derivatives or composites of MXene can dramatically alter some of the material's inherent properties. For instance, MXene nanoribbons exhibit high mechanical properties, with tensile strength as high as 600 MPa and fracture stress greater than 5 GPa, while other 2D materials show better durability. Quantum dots of MXene samples have high strain-at-failure values of more than 10% and are very flexible, meaning they are elastic and ideal for use in nanomechanical applications. Furthermore, the remarkable enhancement of overall mechanical properties in MXene-polymer composites results in high energy absorption values, increasing to more than 100 J/m², and an increase in ductility, with elongation at break reaching up to 30–50% compared to pure MXene. As Khazaei *et al.*^[11] assessed, these advancements make MXenes suitable for applications that demand high mechanical toughness and flexibility.

MXene has low coefficients of thermal expansion, high coefficients of thermal conductivity, and thermal stability. It is, therefore, possible to control thickness, separation distance, or surface coats, which affect these thermal characteristics. For instance, the thermal expansion coefficient of random composites such as MXene-carbon nanotube structures is higher than that of pure MXene structures, and the thermal stability of MXene nanoflowers is higher than that of MXene nanosheets. Additionally, MXene terminated with oxygen (O) and hydroxyl (OH) groups have lower thermal conductivity than those terminated with fluorine (F).^[12]

MXene's chemical properties are characterized by excellent hydrophilicity and chemical reactivity but limited air stability and corrosion resistance. The composition, structure, or surface chemistry of MXene derivatives and composites can further influence these properties. For instance, the oxidation states and the electronic configurations of MXene depend on the type of transition metals in the inclusion. The MXene nanoribbons are more reactive than MXene nanosheets as they possess a higher edge-to-area ratio. Furthermore, the resulting MXene-polymer composites generally exhibit enhanced corrosion protection performance and minimally deteriorate in aerial environments compared to pure MXene.^[11,13]

MXene has a low band gap and resistance but exceptional electrical conductivity and capacitance. These properties can be further tuned by increasing or decreasing layer numbers, changing stacking sequences, or doping. For instance, MXene doped with nitrogen (N) or boron (B) reveals greater electrical resistance than when doped with carbon (C). Dimers like AB stacking have a slightly lower band gap than AA stacking, and MXene with fewer layers has higher conductivity and capacitance than a multilayered structure.^[14]

Regarding magnetic characteristics, MXene possesses

strong spin polarization and magnetoresistance but relatively low magnetic moments and anisotropy. It is also possible to modify the magnetic characteristics by changing the positions of metal atoms, adding vacancies, or applying external fields. However, metal vacancies in MXene structures result in improved spin polarization and magnetoresistance, whereas applying electric or strain fields leads to enhanced magnetization. Furthermore, the inclusion of elements like Mn or Fe results in a higher value of magnetic moment and anisotropy in comparison to elements like Ti or V.^[12,14] It turns out that the changes in layers and the replacement of metal components are essential for the control of the characteristics of MXene and its variants. Such flexibility demonstrates that MXene has virtually limitless uses in many industries.

3. Results and discussion

3.1 The difference between MAX phase and MXenes

MXene is the two-dimensional material obtained from the MAX phase through the selective removal of the A element, usually from group 13 or 14, as Kumar *et al.* (2020) discussed.^[15] MAX phases are 3D layered materials. The change from MAX phase to MXene requires the etching of the A-group layer, usually by applying acids or fluoride-containing solutions, leading to the formation of a 2D material with a superior surface area and modifiable characteristics.^[16]

MXenes have the general formula $M_{n+1}X_nT_x$, where T_x represents surface terminal functional groups that are produced during etching. These covalent functional groups also play a role in determining the electrical conductivity, hydrophilic nature, and chemical reactivity of the resulting MXene. From the most recent studies,^[17] It was discovered that MXenes possess good electrical conductivity, thermal stability, and intercalation of ions; this makes the compounds ideal for industrial uses in many fields such as energy storage, catalysis, water filtration, and electromagnetic shields.

MAX phases are phase substitutions of both ceramics and metals - high stiffness and hardness, high thermal and electrical conductivity, workability, and damage tolerance.^[7] MAX phases possess a hexagonal crystal structure in which A-group elements are located at the centre of edge-sharing deformed X-M6 octahedra where they are interleaved. MXenes have a relatively lower MXene layer structure thickness and high mechanical flexibility from the absence of A-group element in the layers.

MAX phases, the precursors for MXene synthesis, are generally produced using various techniques. Ball milling and high-temperature sintering are among the most commonly applied synthesis methods, where great attention must be paid to the starting materials, their chemical compositions, and sintering parameters. Fluctuations in these aspects directly impact the quality of the synthesized MAX phase and, by extension, the extracted MXene.^[18] For example, the selection of elemental powders and their quality, the initial composition ratio, and the milling conditions (including speed, typically between 300 and 500 rpm, and milling time, up to 48 hours)

influence the formation of well-ordered MAX phases with minimal impurities. The sintering process, conducted at temperatures between 1200 and 1600 °C, contributes significantly to developing dense MAX phases with distinct crystal structures. A 'controlled atmosphere,' such as Argon or vacuum, synthesizes chemical compounds and avoids oxidation.

These variations directly influence the morphology and dimensions of the MXene obtained after the selective etching of the A layers. Sarycheva and Gogotsi found that due to variations in MAX phase quality, cracks are also present in the MXene sheets, which have a layer thickness of about 1-2 nm and lateral sizes ranging from a few microns to tens of microns.^[14] As a result, the efficiency of MAX phase synthesis remains a priority in obtaining MXenes for innovative applications.

The structural transition from the MAX phase to MXene offers various compositional modifications by adjusting the M, A, X, and T components and changing the number of layers and stacking order. This adaptability allows for targeted property optimization to suit specific applications. For instance, among the most researched MXenes, $\text{Ti}_3\text{C}_2\text{T}_x$ has been confirmed to be an excellent candidate for supercapacitor electrodes due to its high capacitance and swift charge-discharge characteristics. Further improvements in synthesis methods have also enabled the development of novel MXene structures, which expand their potential uses in sensor technology, biomedical engineering, and environmental cleanup.^[19] Additional studies have focused on the stability of MXenes under different conditions, with surface terminations affecting their oxidation behavior and chemical stability.^[20]

In summary, the change from MAX phases to MXenes is achieved through structural transformations that change the material properties, producing materials with boundless potential for use. Customizing constituents, surface characteristics, and the architecture of layers opens a world of possibilities for future applications.

3.2 Derivatives of MXene and composites

Quantum dots, denoted as $\text{Ti}_3\text{C}_2\text{T}_x$, are zero-dimensional variants of MXene with nanoscale size and quantum confinement features. They can be produced by exfoliating $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheets in water using sonication.^[8] For example, TiO_2 quantum dots (QDs) are uniformly dispersed on the MXene surface, with a size of approximately 3 nm. This composite can efficiently trap polysulfides and accelerate the conversion of Li_2S ,^[9] thereby improving the electrochemical performance of lithium-sulfur batteries.

In the nanospace between $\text{Ti}_3\text{C}_2\text{T}_x$ MXene and g- C_3N_4 , FeOOH QDs are dual-confined within this composite. The FeOOH QDs exhibit strong pseudocapacitive behavior and are approximately 5 nm in size. This composite can create high-voltage supercapacitors due to its high specific capacitance of 391.78 F cm^{-2} .^[10]

Co QDs are supported on $\text{Ti}_3\text{C}_2\text{T}_x$ MXene nanosheets in

this composite, with the Co QDs being roughly 2 nm in size and bound to the MXene surface through van der Waals interactions. This configuration enhances the composite's properties, making it suitable for applications such as high-performance zinc-air batteries.^[21] The improved catalytic activity for the oxygen reduction reaction (ORR) arises from the synergistic interaction between the highly conductive MXene and the active Co QDs, which aligns with advancements in zinc-ion battery technology reported by Shi *et al.*^[22]

Nanoribbons/MXene composites are produced by mixing MXene nanosheets with nanoribbons formed from various materials. With their unique quantum confinement effects, quasi-one-dimensional nanoribbons can improve MXene's mechanical, optical, electrical, and magnetic characteristics.^[8] Graphene nanoribbons are covalently grafted onto $\text{Ti}_3\text{C}_2\text{T}_x$ MXene nanosheets to create this composite. Compared to pure $\text{Ti}_3\text{C}_2\text{T}_x$ MXene, it exhibits superior electrochemical performance, high specific capacitance, and outstanding rate capability.^[23] With $\text{Ti}_3\text{C}_2\text{T}_x$ MXene nanosheets, boron nitride nanoribbons are grown in situ to create this composite. Compared to pure $\text{Ti}_3\text{C}_2\text{T}_x$ MXene, it offers improved electrical insulation, mechanical flexibility, and thermal conductivity.^[23] A solvent combines the nanosheets of MXene with carbon nanotubes to create this composite when measured against pure $\text{Ti}_3\text{C}_2\text{T}_x$ MXene. It exhibits improved mechanical characteristics, electromagnetic shielding, and microwave absorption. MXene nanosheets are 2D variants of MXene with a lateral scale of several micrometers and a few nanometers in thickness.^[24] Hydrofluoric acid or other etchants can selectively etch the A layers (like Al) from the MAX phases (like Ti_3AlC_2) by intercalation and delamination.^[8] The intercalation of graphene sheets between MXene nanosheets creates this composite. In comparison to pure MXene nanosheets, it offers improved mechanical strength, thermal stability, and electrical conductivity.^[12] Aniline monomers are polymerized in situ on the surface of MXene nanosheets to create this composite. It also exhibits better specific capacitance, rate performance, and cycling stability than pure MXene nanosheets.^[25] Furthermore, when MXene nanosheets are combined with carbon nanotubes in a solvent, the resulting composite shows improved mechanical properties, electromagnetic shielding effectiveness, and microwave absorption capabilities over the pure MXene nanosheets.^[26]

MXene nanosheets/aerogels are made by adding different additives, including polymers, metals, or inorganic nanoparticles, to MXene nanosheets and freeze-drying them. Compared to pure MXene nanosheets, it possesses a customizable surface chemistry, low density, and high porosity.^[27] A new MnO_2 /MXene composite is made by electrostatic self-assembly and is used as an electrode to improve the performance of supercapacitors. The measured voltages of the nanosheets are -23.5 and +26.1 mV. In this case, the MnO_2 's zeta potential changed from a negative value to a positive value. Gem may increase the charge density and

surface activity of the MnO_2 nanosheets. Then, the MnO_2 zeta potential result shows that Gem is securely attached. Additionally, the MnO_2 nanosheets' thin layered structure can be witnessed. Advanced aqueous zinc-ion batteries using 3D high-density $\text{MnO}_2/\text{MXene}$ microflowers. Creation of $\text{MnO}_2/\text{MXene}$ Composites with low Crystallinity for Capacitive Deionization. $\text{MnO}_2/\text{MXene}$ composites that are porous and hierarchical for high-performance supercapacitors.^[27]

$\text{Ti}_3\text{C}_2\text{T}_x/\text{polymer}$ composites are MXene composites in which polymer matrices such as polyvinyl alcohol, polyethylene oxide, or polyaniline are mixed with $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheets. $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheets give these three-dimensional MXene derivatives to their flower-like morphology. It can be hydrothermally treated with sodium hydroxide to create them.^[8] N-doped $\text{Ti}_3\text{C}_2\text{T}_x$ is a derivative of MXene in which part of the carbon atoms in the $\text{Ti}_3\text{C}_2\text{T}_x$ lattice have been replaced with nitrogen atoms. $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheets can be annealed with urea at a high temperature to create it synthetically. These nanosheets can be made by combining them with powdered or liquid polymers and then casting, drying, or electrospinning the mixture.^[8] Composites of MXene that mix $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheets with oxide nanoparticles such as TiO_2 , ZnO , or Fe_2O_3 are known as $\text{Ti}_3\text{C}_2\text{T}_x/\text{oxide}$ composites. They can be produced via electrostatic self-assembly of $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheets with oxide precursors or nanoparticles, hydrothermal treatment, or sol-gel method.^[28]

$\text{Ti}_3\text{C}_2\text{T}_x/\text{carbon}$ nanotube composites combine carbon nanotubes, such as single-walled, multi-walled, or functionalized carbon nanotubes, with $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheets. They can be produced by carbon nanotube-assisted in situ development of $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheets, vacuum filtration, or ultrasonication.^[28] $\text{Ti}_3\text{C}_2\text{T}_x/\text{graphene}$ composites combine graphene sheets, such as functionalized graphene, reduced graphene oxide, or graphene oxide with $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheets. They can be produced by electrostatically self-assembling $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheets with graphene sheets, mechanical mixing, or hydrothermal treatment.^[8] $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheets combined with metal nanoparticles (Au, Ag, Pt, or Pd) are incorporated into MXene composites. They can be produced by electrochemically depositing metal nanoparticles or precursors on $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheets, chemical reduction, or microwave irradiation.^[28] Some MXene composites exhibit various multilayer structures. For example, the surface sites of a 3×3 supercell of Ti_3C_2 and V_2C are randomly occupied with ^-O , ^-F , and ^-OH groups to model the effects of mixed terminations on an MXene layer, as illustrated in the work. This is done to ensure that, when amounted to over nine different supercells, the mixtures repeat themselves an experimentally discovered common ratio of surface terminations. In contrast, the ending atoms for V_2CT_2 are positioned exactly above the V atom on the opposing surface, and they are placed at the hollow sites directly above the central Ti atom for $\text{Ti}_3\text{C}_2\text{T}_2$.^[28]

Table 1: Comparison of characteristics of the MXene and their derivatives.

No	Types	MXene	Surface area (m^2/g)	Applications	Raw material	Structure	Synthesis method	Unique properties	Ref.
1		Ti_3AlC_2 (Al MXene)	10 – 200	Energy Storage, Sensors	MAX Phase: Ti_3AlC_2	Layers of Ti_3AlC_2 with terminations		Excellent electrical conductivity, biocompatibility (for some applications)	[29, 30]
2	Aluminum (Al) and Silicon (Si) based MXenes	Ti_3SiC_2 (Si MXene)	10 – 150	Batteries, Sensors, Catalysis	MAX Phase: Ti_3SiC_2	Layers of Ti_3SiC_2 with terminations	Selective etching, delamination, surface functionalization	Biocompatibility, potential in biosensing and biomedical applications	[31]
3		Ti_2CT_x (Ti Carbide MXene)	20 – 400	Batteries, Supercapacitors	MAX Phase: Ti_2AlC	Layers of Ti_2CT_x with terminations		Excellent electrical conductivity, high capacitance	[32]
1	Carbon/Nitrogen (C, N)-based MXenes	Ti_3CN (Ti Carbonitride)	20 – 300	Sensors, Photocatalysis	MAX Phase: Ti_3AlCN	Layers of Ti_3CN with terminations	Selective etching, delamination, surface	Photocatalytic activity, potential in gas sensing	[33]

No	Types	MXene	Surface area (m ² /g)	Applications	Raw material	Structure	Synthesis method	Unique properties	Ref.
2		V ₂ CT _x (V Carbide MXene)	20 – 200	Batteries, Sensors	MAX Phase: V ₂ AlC	Layers of V ₂ CT _x with terminations	functionalization	High electronic conductivity, potential in sensor applications	[32]
1		Ti ₃ C ₂ T _x (Titanium MXene)	10 – 200	Energy Storage, Catalysis, Sensors	MAX Phase: Ti ₃ AlC ₂	Layers of Ti ₃ C ₂ T _x with terminations		Excellent electrical conductivity, good electrochemical performance	[29]
2		V ₂ CT _x (Vanadium MXene)	20 – 200	Energy Storage, Spintronics	MAX Phase: V ₂ AlC	Layers of V ₂ CT _x with terminations		Unique electronic and magnetic properties	[34]
3	Transition Metals (Ti, V, Cr, Mo, Nb)-based MXenes	Cr ₂ CT _x (Chromium MXene)	50 – 200	Spintronics, Catalysis	MAX Phase: Cr ₂ AlC	Layers of Cr ₂ CT _x with terminations	Selective etching, delamination, surface functionalization	Unique magnetic properties, potential for spintronic applications	[29]
4		Mo ₂ CT _x (Molybdenum MXene)	30 – 300	Energy Storage, Catalysis	MAX Phase: Mo ₂ GaC	Layers of Mo ₂ CT _x with terminations		High electronic conductivity, good electrochemical performance	[35]
5		Nb ₂ CT _x (Niobium MXene)	30 – 200	Energy Storage, Catalysis	MAX Phase: Nb ₂ AlC	Layers of Nb ₂ CT _x with terminations		Good electrochemical performance, potential for energy storage	[36]

From Table 1, MXenes of different compositions like aluminum-, silicon, and transition metal-based, amongst others, have unique characteristics that qualify them for energy storage, catalysis, and sensing. Depending on their structure, synthesis methods, and surface functionalization, they have dramatically different performance characteristics. This means that the given characteristics are adjustable, and proper modifications make MXenes suitable for various applications and technologies in terms of conductivity and electrochemical properties, among others.^[29-36]

3.3 Synthesis methods

MAX phases, nanolaminate ternary carbides, and nitrides possess key characteristics such as high thermal stability,

electrical conductivity, and mechanical strength.^[37] The synthesis techniques for MAX phases can be broadly categorized into solid-state synthesis, self-propagating high-temperature synthesis (SHS), and chemical vapor deposition (CVD).^[38] The main aspects of these methods are discussed below.

Solid-state synthesis is the most common method for producing MAX phases.^[39] This method involves mixing elemental or compound precursors (*e.g.*, Ti, Al, and C powders) in stoichiometric ratios, followed by high-temperature sintering.^[40] For example, the synthesis of Zr₂AlC involves mixing Zr, Al, and graphite powders, pressing them into pellets, and heating them in an argon atmosphere at 1400 °C for 4 hours.^[39] Gonzalez-Julian also noted that the type of

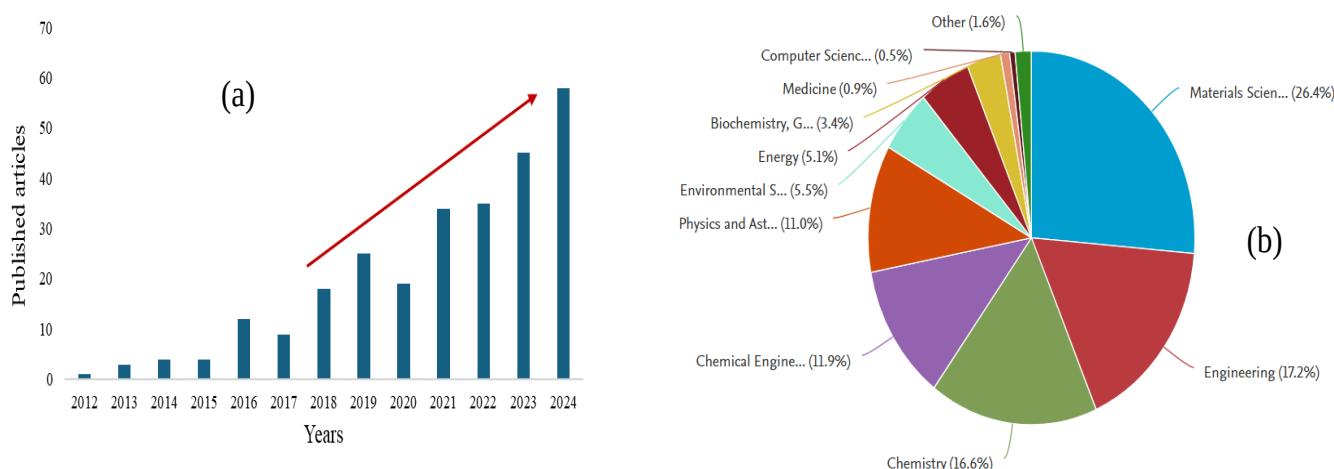


Fig. 2: (a) Growing interest in recent years and (b) a wide range of areas and explored applications.

sintering process plays a critical role, with hot pressing or spark plasma sintering (SPS) providing better phase purity due to densification and reducing the negative effects of by-products. Sintering temperatures typically range from 1200 to 1600 °C, depending on the composition of the MAX phase.^[41]

Self-propagating high-temperature synthesis (SHS) involves an exothermic reaction that allows for the rapid synthesis of MAX phases. The process begins with ignition, propagating through the powder mixture as a combustion wave.^[41] This method is especially useful for quickly producing large amounts of material. In a recent study, Fu and Xia concluded that the reactant ratios and initial pressures are key factors that can be adjusted to control the combustion temperature and wave velocity. For instance, synthesizing Ti_3SiC_2 requires precise Ti:Si:C ratio adjustment to obtain a single phase without secondary phases such as TiC.^[40]

Although less common, chemical vapor deposition (CVD) is an effective method for synthesizing thin films of MAX phases. In this method, precursors are vaporized and transported to a substrate, which reacts and deposits as a thin layer.^[41] Mathis and colleagues demonstrated a modified CVD technique to synthesize environmentally stable Ti_3C_2 by optimizing precursor ratios and deposition temperatures.^[42]

Comparing these methods, it can be stated that solid-state synthesis offers scalability and cost-effectiveness but may require post-synthesis treatments to improve purity. SHS is energy-efficient but requires careful reaction control to avoid incomplete reactions. On the other hand, CVD synthesis is associated with the formation of high-purity thin films but is limited to small-scale synthesis. The choice of method depends on the desired material characteristics and specific applications. Modifying these synthesis methods can lead to the creation of specific MAX phases that are suitable for various industrial and research applications.

3.3.1 MXene synthesis

MXenes are 2D nanomaterials with outstanding electrical, chemical, physical, mechanical, and electrochemical properties. The morphology and unique characteristics of pure

MXenes are largely dependent on the synthesis techniques and working parameters. Since the first synthesis of pure MXenes in 2011, a wide range of synthesis and preparation approaches have been developed, from laboratories to industrial-scale.

MXene synthesis typically involves selective etching of the A-layer from MAX phases, with chemical etching being the most common method. In this process, MAX phases are chemically etched using strong acids such as hydrofluoric acid (HF) or a mixture of hydrochloric acid (HCl) and fluoride salts to dissolve the A-layer of the material selectively. Deen *et al.* suggested that HF concentrations typically range from 10% to 50%.^[43] For instance, the process of producing $\text{Ti}_3\text{C}_2\text{T}_x$ involves immersing Ti_3AlC_2 in a 40% aqueous HF solution at room temperature for 24 hours.^[44] This process produces stacked, multi-layered MXenes, which can be exfoliated into individual layers by ultrasonication in water or organic solvents. The resulting MXene flakes have high electrical conductivity ($10,000 \text{ S}\cdot\text{cm}^{-1}$) and a surface area of $200 \text{ m}^2\cdot\text{g}^{-1}$.

Electrochemical etching is a relatively recent approach that offers greater environmental safety and control. In this process, an electric potential is applied to MAX-phase electrodes in a liquid electrolyte, causing the dissolution of the A-layer. Patil *et al.* reported a three-electrode system with Ti_3AlC_2 as the working electrode in a fluoride-containing electrolyte.^[45] This method allows for better control over etching parameters (voltage and time), synthesizing high-quality MXenes with fewer defects.

Another novel method used for MXene synthesis is molten salt etching, where MXenes are immersed in molten salts, such as LiF or NaCl, and subjected to high temperatures. Awan *et al.* showed that heating Ti_3AlC_2 at 550 °C in molten LiF for 5 hours selectively etches the Al layer, yielding MXenes with improved thermal stability and controlled surface terminations.^[46] This method generates fewer hazardous waste products compared to chemical etching.

In conclusion, chemical etching is an efficient and cost-effective method for mass production, although it involves potentially hazardous chemicals and requires proper safety measures. This method is highly selective for etching but has

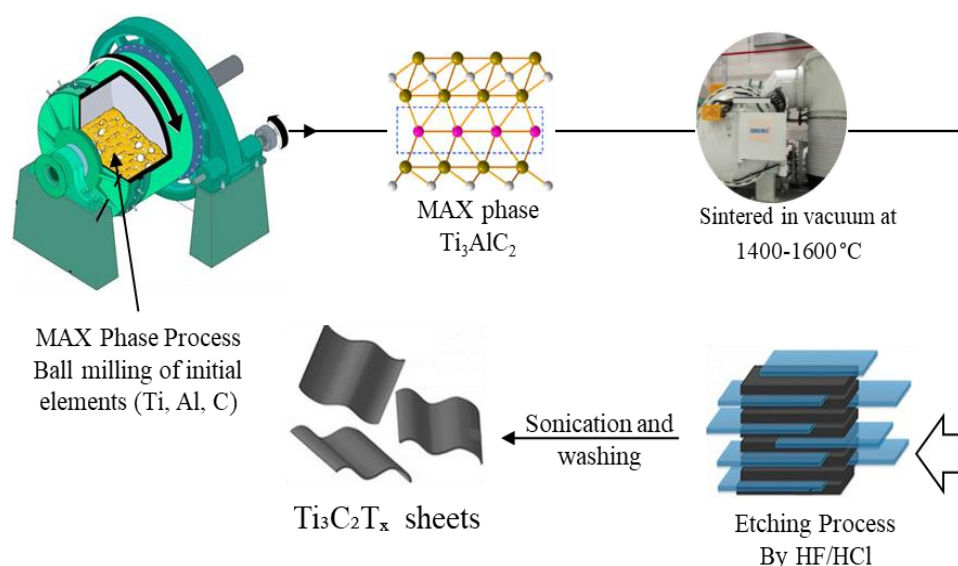


Fig. 3: Experimental setup for synthesizing MAX phases and $\text{Ti}_3\text{C}_2\text{T}_x$ MXenes.

a slower rate for large-scale manufacturing. On the other hand, molten salt etching allows for the fabrication of products with high thermal stability but requires precise temperature control.^[46] These various synthesis techniques help regulate the synthesis of MXenes for different functions depending on their structure and chemistry.^[47,48]

Deen *et al.*^[43] and Murugesan *et al.*^[44] obtained high-yield Ti_3C_2 MXene sheets with a high surface area using HF as the etching agent. The synthesis of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene involves several steps, such as etching, segregation, exfoliation, and functionalization (as shown in Fig. 3). Controlling these processes is crucial to obtaining the desired properties of the final product. The preparation typically involves mixing high-purity titanium, aluminum, and carbon in a stoichiometric ratio of 3:1:2 via ball milling, usually at 300–500 rpm, for 10–24 hours to achieve homogeneity. The resulting mixture is sintered in vacuum or argon at temperatures of 1400–1600 °C for 2–4 hours to create dense crystalline structures of the MAX phase.

The next step involves selective etching of the aluminum (A) layer from the MAX phase to synthesize $\text{Ti}_3\text{C}_2\text{T}_x$. This usually involves a 10–50% HF solution or a safer LiF/HCl pre-impregnation method. For example, etching with 1g of LiF in 20 mL of a solution containing 9 mol/L HCl at 35 °C for 24 hours effectively removes the Al layer while causing minimal damage to the structure. After etching, the product is washed with deionized water until the pH of the resulting substance reaches 6, removing any residual acids and by-products.

After etching, intercalation agents such as dimethylsulfoxide (DMSO) or urea are inserted to increase the interlayer gap, allowing for more flexibility and ion mobility. This step usually takes 12–24 hours and involves ultrasonic dispersion of the etched MXene in the intercalating agent. Finally, the material is freeze-dried or vacuum-dried at temperatures below 50 °C to preserve the layered structure and

surface chemistry, primarily consisting of (-OH, -F, -O). The synthesized $\text{Ti}_3\text{C}_2\text{T}_x$ MXenes exhibit impressive characteristics, such as fully reversible high capacitance (500 $\text{F}\cdot\text{g}^{-1}$) due to their very large surface area and short ion diffusion paths. These characteristics make them ideal for supercapacitor electrodes, as Sarycheva and Gogotsi pointed out, with synthesis parameters significantly impacting their efficiency.^[19]

Furthermore, Zhang *et al.*^[49] demonstrated the synthesis of Ti_3C_2 MXene employing the two-step etching process. The first step entails washing Ti_3AlC_2 with low-concentration HF, and the second step involves etching with different concentrations of FeCl_3 . The material synthesized from the modified method above provided better results than the non modified method of MXene for its application in the electrode material for the supercapacitors. While these improvements have demonstrated some effectiveness, the influence of composite or hybrid structures on material properties is well understood. They generally yielded superior characteristics than individual, unpure materials. In addition, incorporation of MXenes into other materials helps overcome this problem and improve the overall functional characteristics of materials. Table 2 lists the comparison of the synthesis methods of MXene.^[44,50-59]

To mitigate MXene aggregation, researchers have integrated MXenes with other materials, such as carbon-based materials, polymers, and metal oxides. Carbon materials are versatile and exist in various nanostructures like carbon nanotubes (CNTs), CNT fibers, graphene hydrogels, graphene films, carbon fibers, carbon black, and porous carbon. MXene/carbon composites offer a stable support structure that separates individual MXene layers, enhancing ion accessibility and resulting in composites with improved electron transport, larger surface areas, and superior electrochemical properties. Control over the MXene's

Table 2: Significant comparison of the synthesis methods of Mxene.

No.	Methods	MAX phase	Etching agent	Structure	Surface area m^2g^{-1}	Yield (%)	Properties	Applications	Ref.
1	HF etching	Ti_3AlC_2	HF Acid	$\text{Ti}_3\text{C}_2\text{T}_n$	20-30	70-80	High surface area, good conductivity	Supercapacitors, energy storage	[44]
2	In Situ HF generation	Ti_3AlC_2	HF generated via salts	$\text{Ti}_3\text{C}_2\text{T}_n$	25-35	80-85	Environmentally safer, controlled morphology	Supercapacitors, catalysis	[50]
3	Electrochemical etching	Ti_3AlC_2	Electrolyte voltage	$\text{Ti}_3\text{C}_2\text{T}_n$	30-40	85-90	Tunable surface termination, high purity	Energy storage, catalysis	[51]
4	Alkaline etching	Ti_3AlC_2	NaOH or KOH	$\text{Ti}_3\text{C}_2\text{T}_n$	15-25	60-70	Fluorine-free, moderate stability	Catalysis, water purification	[52]
5	Molten Salt etching	Ti_3AlC_2	Low-temperature salts	$\text{Ti}_3\text{C}_2\text{T}_n$	40-50	90-95	High efficiency, fast synthesis	Li-ion batteries, optoelectronics	[53]
6	Mechanical exfoliation	Ti_3AlC_2	Shear forces	$\text{Ti}_3\text{C}_2\text{T}_n$	10-15	50	Simple process, low yield	Prototyping, initial characterization	[54]
7	Inorganic exfoliation	Ti_3AlC_2	Acid intercalation	$\text{Ti}_3\text{C}_2\text{T}_n$	20-30	70-75	Enhanced interlayer spacing	Catalysis, water purification	[55]
8	Organic exfoliation	Ti_3AlC_2	Organic solvents	$\text{Ti}_3\text{C}_2\text{T}_n$	25-25	75-85	Improved dispersion in organic matrices	Polymer composites, electronic devices	[56]
9	Intercalation + exfoliation	Ti_3AlC_2	Organic/inorganic agents	$\text{Ti}_3\text{C}_2\text{T}_n$	30-40	80-90	Excellent control over morphology and structure	High-performance batteries, supercapacitors	[57]
10	Supercritical etching	Nb_4AlC_3	H_2O -assisted supercritical	$\text{Nb}_4\text{C}_3\text{T}_n$	50-60	95-98	Exceptional uniformity, eco-friendly	Energy storage, electronic devices	[58]
11	Hydrothermal	Ti_3AlC_2	Water temperature	$\text{Ti}_3\text{C}_2\text{T}_n$	20-30	85-90	Sustainable, fluorine-free synthesis	Proton supercapacitors	[52]
12	Etching galvanic reaction	Ti_3AlC_2	Etching agents + reaction	$\text{Ti}_3\text{C}_2\text{T}_n$	25-35	80-85	Improved environmental compatibility	Al removal, industrial applications	[59]

synthesis and alteration is crucial since synthesis procedures can influence the outcome of the products' morphology and termination, ultimately impacting the product's performance. For instance, mechanical force can induce changes in electrical properties during micro- and nanoscale formation, and a multilayered structure is desirable for biosensors that carry enzymes to promote faster diffusion of target molecules.^[60] In the following sub-sections, the different

synthesis methods that can be employed to produce MXene will be discussed.

The data in Table 3 reveals the great potential of MXenes, particularly $\text{Ti}_3\text{C}_2\text{T}_x$, as supercapacitor electrodes due to their versatility and the variety of methods of preparing and modifying the material in terms of specific capacitance.^[15,16,61-79] These fluctuations admit the role of synthesis techniques and structural engineering in improving MXene performance.

Table 3: Comparison of the synthesis methods of the MXene to specific capacitance.

Material	Methods	Specific capacitance	Ref.
Ti ₃ C ₂ T _x	Selectively etching	1.5 F cm ⁻³	[61]
Ti ₃ C ₂ T _x	Brade coating, and laser scribing	241 (5 mV/S) F/g	[62]
Ti ₃ C ₂ T _x ionogel	Assimilation into the polymer	61 (25 μ A/cm ²)	[63]
Nb ₄ C ₃ T _x	HF etching Nb ₄ AlC ₃ and TMAOH delamination	1075 Fcm ⁻³	[64]
V ₂ NT _x	Selective etching	112.8 F/g	[65]
Ti ₃ C ₂ T _x	Selective etching	427 F/g	[16]
Ti ₃ C ₂ T _x	Selective etching	61 F/g	[66]
Ti ₃ C ₂	Hydrothermal method with microwave radiation	418.07 F/g	[67]
Ti ₃ C ₂ T _x - NOMC hybrid	Selective etching of the Al layer from MAX (Ti ₃ AlC ₂)	329 (1 A/ g) F/g	[68]
MXene			
MXene/CNT paper	Alternating filtration method	150 (2 A/ g) F/g	[69]
MXene/rGO	Electrostatic self-assembly of negatively charged MXene and positively charged chemically reduced graphene oxide	335.4 (2 A/ g) F/g	[70]
	Selectively etched with the assistance of LiF and HCl to acquire f-MXene. Subsequently, the MF was immersed in the suspension of Co(SCN) ₂ @MXene		
CoS@MXene/CF		250 (1 A/ g) F/g	[15]
3D porous MXene-rGO	A facile self-propagating approach to fabricate 3D porous MXene-rGO film	329.9 (1 A/ g) F/g	[71]
MXene/N-Carbon	Environment-friendly method, which exists in the form of 3D hollow neuron-like architecture	332 (1 A/ g) F/g	[72]
Ti ₃ C ₂ T _x /Ni-MOFs	Wet-chemical etching in hydrofluoric acid	1124 (1 A/ g) F/g	[73]
NiCoS/d- Ti ₃ C ₂ T _x	In situ sulfuration technique and co-precipitation	758.9 (1 A/ g) F/g	[74]
Carbon-intercalated Ti ₃ C ₂ T _x	Insitu carbonization	364.3~193.3 F/g	[75]
Ti ₃ C ₂ T _x /carbon cloth	Freeze-drying with KOH treatment	312~200 F/g	[76]
Nanoporous Ti ₃ C ₂ T _x film	Freeze-drying and mechanically pressing	932~462 F/cm ³	[77]
N,S-MXene/rGO	Thiourea intercalation between Ti ₃ C ₂ T _x MXene flakes and the doping of sulfur and nitrogen atoms by annealing treatment	698.5 F/cm ³ at 1A/g	[78]
MXene/holey graphene	Hummers method	1445 F/cm ³ at 2 mV/s	[79]

For example, Capacitance values of Ti₃C₂T_x can range between 1.5 Fcm⁻³ to > 1124 Fg⁻¹, depending on the Selective etching or the incorporation of another material into Ti₃C₂T_x.

It was established that adding Ni-MOFs or rGO further enhances the capacitance, mainly due to higher surface area, enhanced conductivity, and cooperative interactions of Ti₃C₂T_x layers with the materials added to the hybrid structure. These hybrids have improved electron transport and ion diffusive properties, both desirable requirements for high efficiency in supercapacitors.

Moreover, the other composites of N, S-MXene/rGO and MXene/holey graphene reveal the importance of doping intercalation and controlled structural change for better electrochemical performance, providing capacitance 698.5 Fcm⁻³ for N, S-MXene/rGO, and 1445 Fcm⁻³ for MXene/holey graphene. Techniques such as freeze-drying, mechanical pressing, and chemical intercalation complement the specific capacitance by increasing the porosity and structural integrity of the material, thus increasing the energy storage. It can be seen from the table that the electrochemical performance of

MXene-based materials has a direct relation with the synthesis approach, post-sulfation treatment of MXene, and the incorporation of other functional materials. Optimization of these aspects can enhance their particular capacitance to the extent of making MXenes a potential candidate for future energy storage systems. Future research should be directed towards improving synthesis on a vaster scale, reducing the process of restacking, and increasing the material's longevity in external conditions to maximize their application within supercapacitors.

3.4 Application in supercapacitors

Supercapacitor (SC) devices are fascinating due to their wide range of applications, which extend from wearable electronics to energy storage in electric vehicles. Choosing the best electrode material for a given application is one of the most critical factors affecting the efficiency of supercapacitors. MXene's excellent properties, such as remarkable biocompatibility, huge interlayer spacing, conductivity, and environmental friendliness, are the reasons for its wide

acceptance.^[62,63] MXenes and their composites offer enhanced mechanical flexibility and stretchability due to structural design, enabling applications in wearable sensors, energy storage, and electromagnetic shielding.

MXene-based electrodes are promising for energy storage because of their unique electrochemical behavior, especially in acidic environments. When used in acidic electrolytes like H_2SO_4 , MXenes exhibit rapid redox reactions and high capacitance due to proton intercalation without significant structural changes. This results in fast energy storage and excellent cycle life (over 10,000 cycles). Their high performance stems from the ability to store charge through a process called intercalation pseudocapacitance, where protons are inserted between MXene layers. While these materials deliver high capacity and fast charging, their use is constrained by a narrow voltage window. Blending MXenes with solid-state electrolytes is another research hotspot focusing on safe and non-leaky, high-energy storage devices for wearable and flexible electronics. In general, it is proven that MXenes have high efficiency and applicability in capacitors and energy storage systems.^[80]

To improve the performance characteristics of electrode materials, vast experimental and research work has been directed towards the synthesis and preparation of MXene-based hybrid composites, including MXene/organic composites,^[81] MXene/polymer systems,^[14,21] MXene/metal-organic frameworks (MOFs),^[23,24] MXene/carbon materials,^[25,27] and so on. Tang *et al.*^[81] investigated the electrochemical properties of the electrode materials synthesized from pure MXenes and MXene/organic composites and concluded that the prepared MXene/organic composites had better electrochemical performance. Fan *et al.*^[76] synthesized a new method for surface modification of MXene films with the in-situ polymerization of polypyrrole microemulsion particles and ionic liquids for improved supercapacitor applications.

With the rising global requirement for enhanced energy storage density, longer cyclability, and lower cost, there is growing research interest in 2D nanomaterials such as MXenes, particularly $\text{Ti}_3\text{C}_2\text{T}_x$, because of their superior electrochemical performance, high electrical conductivity, and chemically tunable surface. However, it can still be aggregated and easily oxidized, which has led to attempts at composites with other 2D nanomaterials, including COFs, TMCs, LDHs, and MOFs. Using nanoarchitectural engineering to incorporate these materials, the performance of MXenes in supercapacitors (SCs) can be enhanced. This approach not only removes current restraints but also opens the door to HPESS technologies with the ability to meet present and future industrial demands.^[18]

Ramachandran *et al.* successfully developed a binder-free supercapacitor electrode material by integrating Co-MOF spheres and Co-MOF/ $\text{Ti}_3\text{C}_2\text{T}_x$ onto Ni foam. Various electrochemical methods were used to assess the influence of pseudocapacitance arising from the interaction between Co-

MOF and $\text{Ti}_3\text{C}_2\text{T}_x$ on Ni foam. The mesoporous electrodes exhibited notable electrochemical performance in a 3M KOH electrolyte solution, achieving maximum gravimetric capacitances of 3741 Fg^{-1} for Co-MOF/ $\text{Ti}_3\text{C}_2\text{T}_x$ @Ni and 2872.5 Fg^{-1} for Co-MOF@Ni.^[82]

$\text{Ti}_3\text{C}_2\text{T}_x$ MXene is a promising material for enhancing supercapacitor applications because of its outstanding properties; however, several questions remain unanswered about how to improve the structural design of these materials. By improving these aspects, we can optimize the application of $\text{Ti}_3\text{C}_2\text{T}_x$ -based SC devices and possibly address problems with current energy storage technologies. The ability of supercapacitors to deliver high-power bursts of energy and their desirable traits, like cycling stability, safety, and power density, make them invaluable. However, their performance is capped by the electrical double-layer capacitance of $\text{Ti}_3\text{C}_2\text{T}_x$ nanomaterials.

It's crucial to develop scalable and cost-effective synthesis methods for $\text{Ti}_3\text{C}_2\text{T}_x$ while improving its conductivity through doping and surface modifications. Another important aim is to solve the problem of sustainable stability and develop the concept of SC devices that can be versatile, thin, and miniaturized for use in multiple fields. Investigating other MXenes that are not based on Ti and the construction of new types of nano heterostructures will expand the horizons of applying MXenes in energy storage. This multifaceted approach ensures $\text{Ti}_3\text{C}_2\text{T}_x$ remains at the forefront of energy technology innovations, bridging gaps in performance and practicality for a range of critical applications.

4. Conclusion and perspectives

The primary focus on improving supercapacitor performance lies in synthesizing and optimizing high-performance electrode materials. This review highlights the advantages and disadvantages of various types of carbon modifications, including activated carbon, graphene, and CNTs, for use in supercapacitor electrodes. However, it is concluded that simply increasing the surface area does not always improve the energy characteristics. These variations are due to the interactions between ions within the electrolyte and the carbon material's pore size and/or surface chemistry, influencing charge storage and transfer processes within the electrode architecture.

MXenes, layered materials derived from carbides and nitrides, have attracted considerable interest in recent years due to their conductivity, stability, and versatility in energy storage and sensing applications. These developments surpass even graphene, particularly through advancements in synthesis, structural design, and proper integration to achieve desired properties. Notably, MXenes show great promise in supercapacitors, serving as highly efficient electrodes for micro- and solid-state devices, with their tunable porosity contributing to enhanced electrochemical performance.

We suggest that the synthesis of carbon materials from various types of waste, including food and plant waste, should

be further optimized to achieve the best pore size distribution, favoring electrolyte migration and charge carrier movement. Consequently, it is anticipated that integrating MXenes with carbon components in electrode configurations will improve capacitance and enhance charging and discharging capabilities. In these hybrid systems, MXenes are expected to contribute additional pseudocapacitance through faradaic reactions, while the carbonaceous components will contribute to charge storage through the electric double layer. This synergistic interaction can potentially significantly improve energy storage efficiency and supercapacitor performance.

MXenes' unique properties position them as leading candidates for next-generation supercapacitors. While significant progress has been made in their synthesis and performance optimization, addressing challenges such as environmental stability, scalability, and electrochemical limitations is essential for widespread adoption. Future studies should focus on multi-scale modeling, advanced characterization, and sustainable manufacturing practices to unlock the full potential of MXenes in energy storage applications.

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Conflict of Interest

There is no conflict of interest.

Supporting Information

Not applicable.

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