BASICS, PROPERTIES, FABRICATION, AND POTENTIAL APPLICATIONS OF MXENES

Abstract

Researchers are attracted to 2D layered materials because of their unusual electrical, chemical, and physical properties. As a result, they have applications in electronics, sensing, energy storage, and catalysis. Over the years, intense research on new 2D materials has been conducted, fueled by graphene's exceptional characteristics. MXenes are a group of 2D materials produced by the chemical delamination of ternary or quaternary layered carbides or nitrides. They are currently among the most promising materials under study. Among the distinguishing features of the MXene family are its complex bonding, atomic stacking, surface terminal groups, and synthesis routes. MXenes remain an understudied class of materials despite their increased interest. In the current work, we go into great depth on the characteristics of MXenes and assess its potential for the creation of biosensors, and energy storage devices. A summary of recent advancements in this quickly evolving topic is given, along with recommendations for potential future study routes.

Keywords: MXenes, graphene’s, 2D materials, synthesis, applications.

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I. INTRODUCTION

MXenes are the fastest-growing class of two-dimensional materials with distinct characteristics and scalable synthesis techniques. They have gained great scientific interest [1,2]. Since the discovery of stable 2D atomic carbon sheet graphene, its advantages over bulk equivalents have prompted research advancements in various fields. Nanotechnology experts believe that 2D materials like graphene, black phosphorous, stratified double hydroxides, and transition metal dichalcogenides have significant potential for use in different industries such as optoelectronics, biomedicine, and energy storage because of their unique optical, electrical, and mechanical properties. Researchers have been exploring the potential of these materials for many years [3-7]. MXenes have variable surface chemistry, metallic conductivity, and a redox potential similar to graphene. MXenes can be utilized for environmental applications since they often contain non-toxic elements like Ti, Si, and N. At Drexel University, titanium carbide (Ti_3C_2) MXene was first discovered [8].

In addition, recently, MXenes have found diverse applications such as electronics, biosensing, environmental remediation, water desalination, sensors, electrodes, optical devices, and quantum dots [9,10]. Electrochemical capacitors (ECs) and metal-ion batteries are among the most promising electrochemical energy storage technologies. MXene has enabled significant advancements in understanding charge-storage processes and creating innovative, ultra-high capacitance MXene electrodes [11]. MXenes have gained interest across scientific fields such as materials science, environmental science, nanotechnology, chemistry, and physics.

In the 2D domain, MXenes are among the most recent transition metal carbides, nitrides, or carbonitrides to appear [12-15]. The formula for these materials is $M_{n+1}X_nT_x$ (n = 1, 2, 3), where M and X represent early transition metals, nitrogen or carbon, respectively. In the same way, we do with surface terminals T_x such as -O, -F, or -OH. A few examples of these transition metals are Scandium, Vanadium, Titanium, Tantalum, Chromium, Zirconium, and Molybdenum. There are three common structures for MXenes (M_2X, M_3X_2, and M_4X_3). MXenes exist in over 200 different stable phases, including Ti_2CT_x, $Ti_3C_2T_x$, Mo_2CT_x, Nb_2CT_x, $Ta_4C_3T_x$, Ti_4N_3Tx, $Cr_2TiC_2T_x$, $Zr_3C_2T_{x,}$ and V_2CT_x. In addition, MXene has excellent surface hydrophilicity and electrical conductivity in addition to its 2D layered structure. It is also possible for MXene to sandwich a variety of cations between its layers. These outstanding characteristics have drawn considerable attention in fields such as electrochemical energy storage, catalysis, gas adsorption, gas sensing, etc [16-18]. In this chapter, we will investigate the fundamentals, properties, fabrication approach, and potential applications of MXenes through several research findings.

II. MAX PHASES: THE PRECURSORS OF MXENES

In the first decades of the 1960s, the MAX phases were found [19]. According to Fig. 1 [1], MAX phases are hexagonal carbides and nitrides with the formula $M_{n+1}AX_n$ (n=1, 2, 3). Generally, "M" indicates transition metals, while "A" indicates major group elements (such as Si, Ge, Al, Ga, In, Tl, Sn, Pb, P, As, Sb, and Bi), and "X" represents carbon/nitrogen. In the Max phase, M-X bonds are stronger than M-A bonds. It is easier to etch away the more chemically reactive A layers to obtain $M_{n+1}X_n$layers, because of their high surface energy these are typically formed by -F, -OH, and -O groups [20]. These ceramic-metal hybrids

exhibit distinctive properties resulting from their uncommon combination of characteristics [21].

A MAX phase has high thermal and electrical conductivities, excellent machinability, and no corrosion resistance, in contrast to a conventional ceramic which has a low density, a high hardness, and no corrosion resistance [22-24]. A MAX step has three parts, according to Fig. 2 [25], 211 for n=1, 312 for n=2, and 413 for n=3). There is a large number of MAX phases in the space group D^4_6h-P_{63}/mmc, which is a two-unit unit space group. These unit cells consist of layers of A elements sandwiched between layers of MX_6 octahedral layers, with X atoms occupying the octahedral spaces between the M atoms [25].

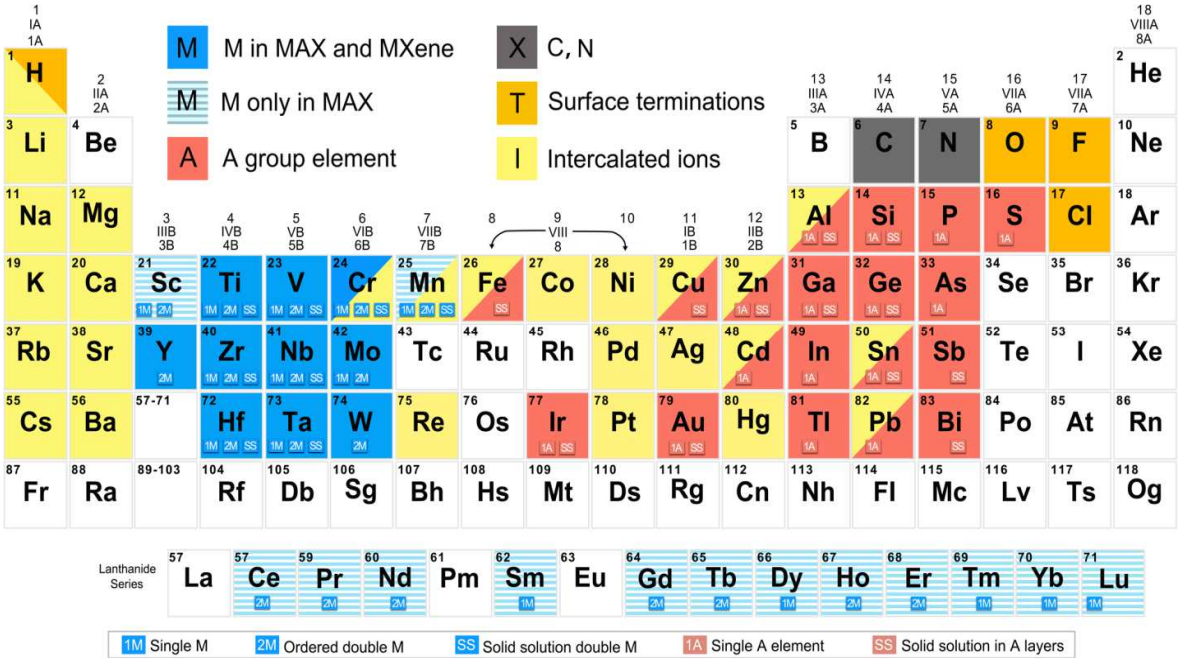

Figure 1: Constituent elements of MAX, MXenes and their intercalation ion. Reproduced with permission[1]. Copyright 2019, ACS Nano

III. PROPERTIES OF MXENES

A few things to keep in mind the kind of MXene precursor used, the etching process, the intercalation method, and the sonication frequency all affect MXene optical, electrical, thermal, mechanical, and magnetic properties shown in Fig. 3. There are numerous and adaptable interactions on the MXene surface due to the electrons linked to transition metal atoms.

1. **Optical Properties:** Gogotsi Y. et al. [26] pointed out that there is little research on the optical characteristics of MXenes. The researchers demonstrated a transmittance of 91.2% for the 5 nm thick $Ti_3C_2T_x$ film, and it can absorb light in the Ultraviolet-Vis range from 300-500 nm [26]. Zhang C. et al. [27] studied that the MXenes concentration in solution determines how much of it is absorbed, whether as a colloidal form or as a thin film. It is the thickness of MXene thin films that causes an increase in absorption when it comes to thin films. At 550 nm, $Ti_3C_2T_x$ MXenes in pure and intercalated forms have transmittances of 77% and 90%, respectively [27]. Wang et al. [28] investigated the

existence of the functional groups (-O, -F, and -OH) on the surface affects the optical characteristics of MXenes. Ti_3C_2 MXenes can function from infrared to ultraviolet light because -F and -OH have low in-plane absorption coefficients. The in-plane absorption coefficients are greater for naked and -O functionalized MXenes. MXenes are hopeful candidates for flexible transparent electrode applications considering their optical transparency and metallic conductivity in the visible region, but their strong reflective properties in the ultraviolet area suggest anti-ultraviolet coatings. Lastly, it demonstrated high light-to-heat conversion efficiency (100%), which is helpful for biological applications [28].

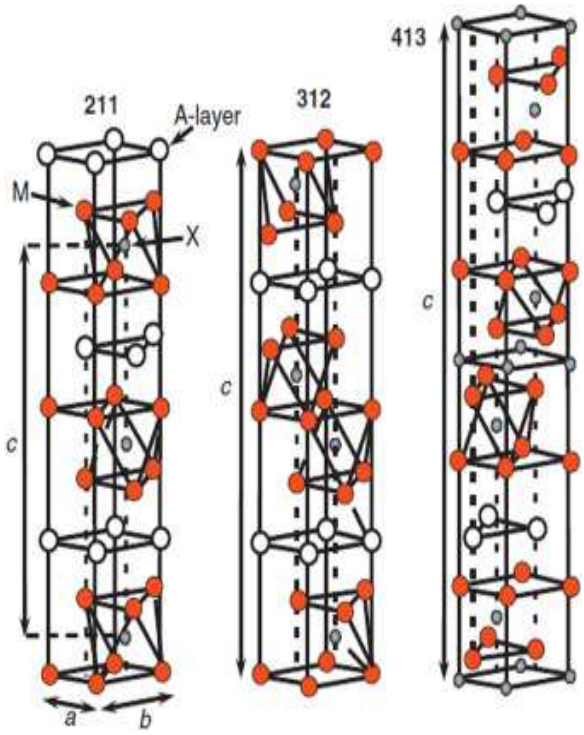

Figure 2: Crystal structure of MAX phases 211, 312, and 413 (*n*=1-3). Reproduced with permission [25]. Copyright 2017, Elsevier

2. **Thermal Properties:** For storage and use in solutions and thin films, it is essential to comprehend the thermal stability of MXene. The thermal stability of MXenes was discovered to be substantially influenced by both their chemical makeup and the environment by combining thermogravimetric and mass spectrometry analyses. According to Toulouseian research, although having a hexagonal structure, $Ti_3C_2T_x$ (T_x = OH or F) is still stable at 800 °C, and its hexagonal structure in the Ar structure is still there even at 500 °C. Thermogravimetric investigations show that $Ti_3C_2T_x$, which turns into TiC in an argon (Ar) atmosphere, has a substantial weight rise at temperatures exceeding 800 °C. $Ti_3C_2T_x$ MXene is partly annealed in an anatase TiO_2 atmosphere at 200°C and fully annealed in a rutile TiO_2 atmosphere at 1000°C. It is possible to create TiO_2 with a variety of crystal structures and morphologies by adjusting the annealing temperature, the pace at which MXene is heated, and the length of the oxidation process. The final result is a wide range of MXene-based hybrids and derivatives. Due to their high surface energy and usual thermodynamic metastability, MXenes with exposed metal atoms on their surface frequently spontaneously oxidize in air [30, 31].

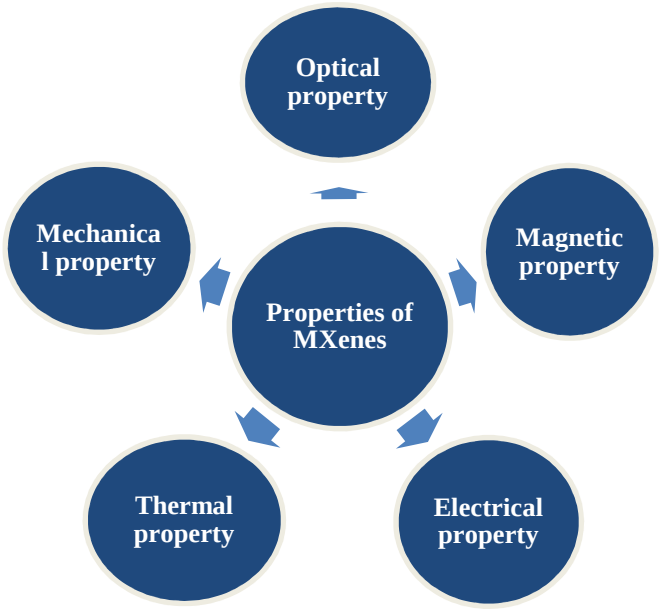

Figure 3: Properties of MXenes

3. **Mechanical Properties:** Regarding the MXenes mechanical characteristics, Section The study is mostly concerned with mathematical computations. There have been several theoretical research on graphene and other 2D materials that resemble graphene, but some of these works are theoretical and experimental investigations on the mechanical properties of MXene material [32]. Because M-C and M-N bonds are among the strongest, mechanical features of MXenes have piqued the interest of researchers. Several studies have validated the mechanical properties. It is noteworthy that as the number of layers increases, the young modulus of MXene carbide and nitride decreases [33]. For 2D materials, evaluating their mechanical properties remains difficult, despite that numerous mechanical testing methods exist for the characterization of bulk materials. Two-dimensional nanomaterial mechanical properties were primarily measured via nanoindentation, which involves delivering force to the center of a 2D material film with an AFM tip. A monolayer Young Modulus of 333 ± 30 GPa was recently obtained using this method [34,35].

4. **Magnetic Properties:** Among the MXene phases, Cr_2CT_x and Cr_2NT_x exhibit good magnetic order over long distances compared with $Ta_{n+1}C_n$, which shows significant long-range ferromagnetism. Magnetic properties of MXene are reduced or eliminated by the activated functional groups. In the MXene family, two-dimensional layers exhibit a wide range of magnetic properties. MXenes 2D Ti_3C_2 and Ti_3N_2 are predicted to be antiferromagnets, while 2D Cr_2C, Cr_2N, Ta_3C_2, and Cr_3C_2 are predicted to be ferromagnets that may dissociate from their MAX phases [36]. Because MXenes phases are possible to have different magnetic characteristics than MAX phases, studies made it possible to assess their magnetic characteristics more widely. Several antiquity-related substances have been predicted to possess magnetic moments, including Ti_4C_3, Fe_2C, Ti_3CN, Zr_2C, Cr_2C, Ti_3N_2, Zr_3C_2 and Ti_2N. Finally, a unique analysis should be conducted for each MXenes and functionality group. The magnetic properties of Ti_3CNT_x and Ti_4C_3Tx are lost when functional groups are added, whereas Cr_2CT_x and Cr_2NT_x with -OH and -F groups maintain their magnetic properties at ambient temperatures, and

Mn_2NT_x is magnetic regardless of surface termination. The reported magnetic moments have yet to be confirmed by experiments; they are based on computer forecasts. This is explained by the poor control of surface chemistry and the insufficient production of MXene molecules [37-40].

IV. FABRICATION OF MXENES

Materials science and nanotechnology have been greatly impacted by MXene since its discovery in 2011. Theoretical calculations predict more than 100 different MXene compositions and more than 40 MXene structures have already been created in the laboratory from top-down approaches or bottom-up approaches. MXenes can be made biocompatible by modifying their surface functions. To get a better idea of the achievements made in the synthesis and modification of MXenes, we can highlight some outstanding reviews [41,42]. The preparation of MXenes is given below in Fig. 4 [42].

1. **Top-down Strategy:** A top-down MXene is produced by selectively removing the A atomic layer from the precursor while leaving the $M_{n+1}X_n$ layers intact. Due to the nominal oxidation state of the A atom in MAX (for example, Al^0 in Ti_3AlC_2), the etching process involves oxidizing it to another state, such as Al^{3+} or Si^{4+}. The strength of the M-A bond in comparison to the M-X bond, as well as other factors such as the Gibbs free energy of by-product formation, affect the etching reaction's Gibbs free energy, which determines the ability to remove the A element in the case of HF etching of Al-based MAX phases. In general, exfoliation involves oxidation of the A element, followed by conversion into soluble by-products via ligation [43]. The most traditional and popular top-down approach to making MXene is HF etching. There are two distinct steps in the interaction between MAX and HF during the etching process. Using Ti_3AlC_2 MAX as an example, Ti_3AlC_2 is fully devoid of Al atoms after being submerged in a 50% (w/v) HF solution for two hours at room temperature. As a result, Ti_3C_2 is produced [44]. The reaction between hydrofluoric acid solution and Ti_3AlC_2 is as follows:

$$Ti_3AlC_2 + 3HF = AlF_3 + 3/2H_2 + Ti_3C_2 \quad \text{(i)}$$

$$Ti_3C_2 + 2H_2O = Ti_3C_2(OH)_2 + H_2 \quad \text{(ii)}$$

or

$$Ti_3C_2 + 2HF = Ti_3C_2F_2 + H_2 \quad \text{(iii)}$$

The same etching technique is used to prepare more MXenes, including Ti_2CT_x, V_2CT_x, and Mo_2CTx. Handling and disposal of HF are hazardous because of its great toxicity and capacity to infiltrate through skin, tissue, and even bones. The usage of HF has been greatly reduced or eliminated through the development of numerous ways to improve the etching process. According to Alhabeb et al., there is a risk of creating hazardous HF when using the in situ approach to create $Ti_3C_2T_x$ over a 24-hour etching time [45]. It was found that as little as 5 wt% HF can still be used to manufacture $Ti_3C_2T_x$. In 2014, Ghidiu et al. etched the conductive "clay" (Ti_3C_2Tx) by replacing the highly corrosive HF with fluoride salts such as NaF, KF, and LiF. To achieve delamination, large organic compounds dissolved in dimethyl sulfoxide (DMSO) or

tetramethylammonium hydroxide (TMAOH) are utilized alongside NH_4HF_2 or HF, followed by sonication. The amount of LiF and HCl used determines whether delamination of $Ti_3C_2T_x$ intercalated with Li+ using HCl-LiF can be done with or without sonication. The in situ technique offers the advantage of creating a larger interlayer space between water, which reduces their interaction with MXene compared to direct HF etching [46].

2. **Bottom-up Strategy:** Instead of starting with bulk material, bottom-up methods use molecular material as a starting point. The transparent MXene films were created by selectively removing the A-element in the first bottom-up synthesis of MAX film. In this first work, they used direct current (DC) magnetron sputtering to deposit thin films of Ti_3AlC_2 onto a TiC layer (which served as an incubation layer) while maintaining an ultrahigh vacuum and a temperature of 780 °C. Transparent, conductive $Ti_3C_2T_x$ epitaxial films with well-maintained metallic conductivity down to 100 K were produced after the aluminum layer was removed using HF or NH_4HF_2 [48].

This approach can be used to create Mxene-derived quantum dots (MQDs) from microscopic precursors of inorganic and organic compounds. Bottom-up methods have several benefits, including better control over the structure and characteristics of quantum dots (QDs), increased atomic use, and faster functionalization. Previous investigations have provided a solid foundation for preparing bottom-up MXene synthesis. The production of large- scale products requires simple, highly efficient precursors with excellent crystallinity, low toxicity, monodispersity, moderate reaction conditions, and high yields. MXene is most likely to be developed to meet incremental application requirements using one-pot bottom-up methodologies in the future because they have simpler operating conditions than top-down approaches [49].

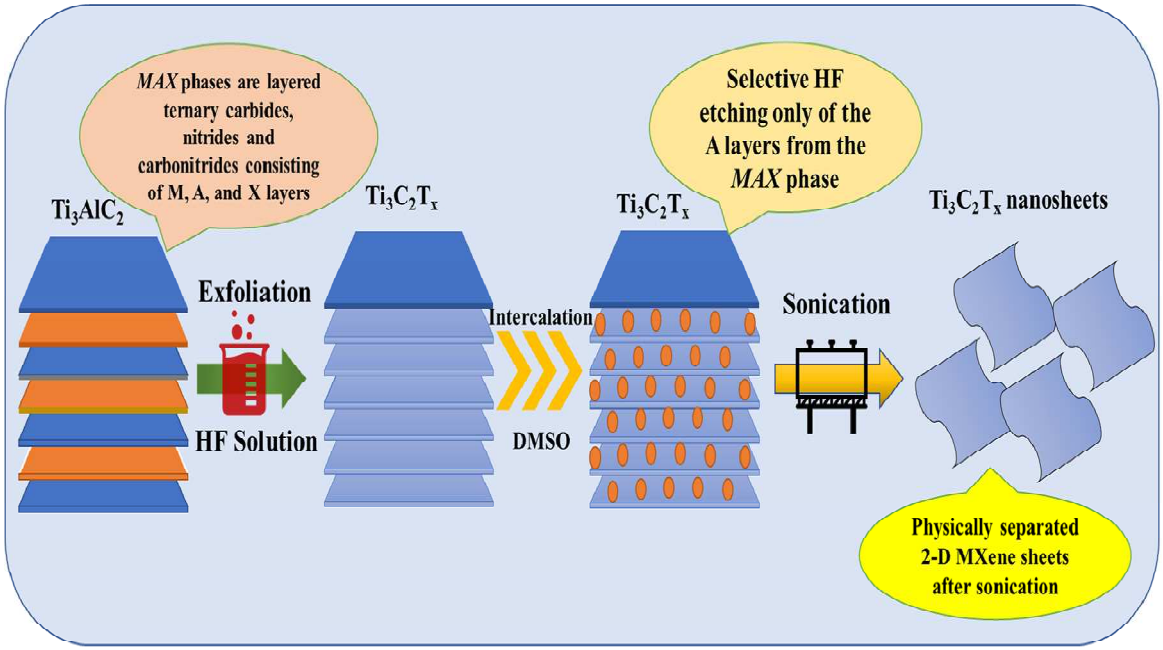

Figure 4: Preparation of MXenes by intercalation method. Reproduced with permission [42]. Copyright 2021, Springer

V. APPLICATIONS OF MXENES

1. **Energy Storage:** Because of the depletion of fossil fuels and the increased consumption of energy, there has been a growing interest in storage and conversion systems, as well as renewable energy sources such as supercapacitors and batteries [50]. Although energy storage was the earliest and most investigated application for MXenes, MXenes have shown significant potential in different types of other applications. Anasori et al. [48] present a detailed assessment of the majority of MXenes energy and nonenergy-related applications researched thus far. First-principles calculations are an important tool in the search for prospective applications due to their cost and time efficiency compared to experimental efforts. MXenes are 2D layers that can contain ions of varying sizes and are useful for non-lithium ion batteries, as electrode material choices are currently limited. Some oxygen-terminated MXenes have theoretical capabilities in Mg, Ca, Na, K, and Al-ion batteries. It's worth noting that Na^+ and other ions are predicted to create an extra metal layer, doubling the capacity. Furthermore, a wide range of functional capabilities can be achieved with MXenes due to their structural and chemical diversity, as well as their ability to tune their surface chemistry. This makes some of them suitable for anodes, while others are suitable for cathodes [51,52].

   Due to their high metallic conductivity, MXenes, particularly Ti_3C_2Tx, are frequently utilized in supercapacitor applications to facilitate rapid electron transfer. It has been discovered that MXene, a 2D material, can store energy through electrochemical or spontaneous interconnection by metal ions [53]. During adsorption in MXenes, ions diffuse to shallow and deep adsorption sites. As a result, they are initially accommodated at the particle's edges at low adsorption sites, and then at deep sites in the particle's center. This results in high ion adsorption activation. Show off your energy. Ions rapidly adsorb at low absorption locations with MXenes, making them the best alternative for precise charge storage quantification and improved rate performance. Additionally, cation intercalation can affect the interlayer water content in MXene nanosheets. These nanosheets precipitate in a partially hydrated form with kosmotropic ions such as Mg^{2+}, Li^+, and Al^{3+}. On the other hand, chaotropic ions like TEA^+ and Cs^+ can successfully dehydrate MXenes. During the process of extracting and inserting ions from aqueous media, there are frequent changes in C-value. It has been observed that the deformation behavior generated by different ions in MXene is different from that of interlayer gap extension in graphite. The interlayer spacing between $Ti_3C_2T_x$ (MXene) nanosheets can be minimized by using cations with high charge and short ionic radius for interpolation while using cations with high ionic radius and low charge will lead to interlayer expansion of MXene [54].

2. **Biosensors:** Electrochemical sensors are the most commonly used biosensors for analysis. This type of sensor offers various advantages for application in biomaterial detection due to its ability to measure observable electrochemical properties based on the properties of the target material that preferentially react with bio-receptors [55]. Biosensing has gained much interest as a good tool for analyzing specific biomolecules in a different types of applications. The investigation of direct electron transfer (DET) between electrodes and enzymes is crucial for making mediator-free electrochemical biosensors [56, 57]. The DET between electrodes and enzymes is limited, however, because proteins' electroactive centers are buried deep within their protein structures, and

the electrode's electrolytes often kill electrode surface proteins. Nanomaterials have been used to immobilize enzymes on electrode surfaces to overcome this problem. Due to their outstanding electrical characteristics, high biocompatibility, and high surface area, MXene-based materials have shown promise as biosensing nanomaterials [58].

3. **Energy Harvesting:** Solar energy is considered a hopeful option to meet our energy needs in the future because of its exceptional characteristics. It is ecologically friendly, unlimited, and safe. Therefore, technologies that can directly convert solar energy into electrical energy are receiving a lot of attention. MXene, a two-dimensional material, has the potential to be used in solar cells. Three types of materials can be applied to it: electron/hole transport layers, electrodes, and other materials [59]. Because $Ti_3C_2T_x$ is the representative member of the MXene group, it offers appropriate flexibility, outstanding electrical conductivity, and high transmittance as solar cell electrodes. This section discusses the use of $Ti_3C_2T_x$ electrodes in sequencing solar cells made of dye-sensitized, perovskite, silicon, and organic materials. MXenes can serve not only as bulk materials for electrodes but also as additives in solar cell components [60].

VI. FUTURE SCOPE

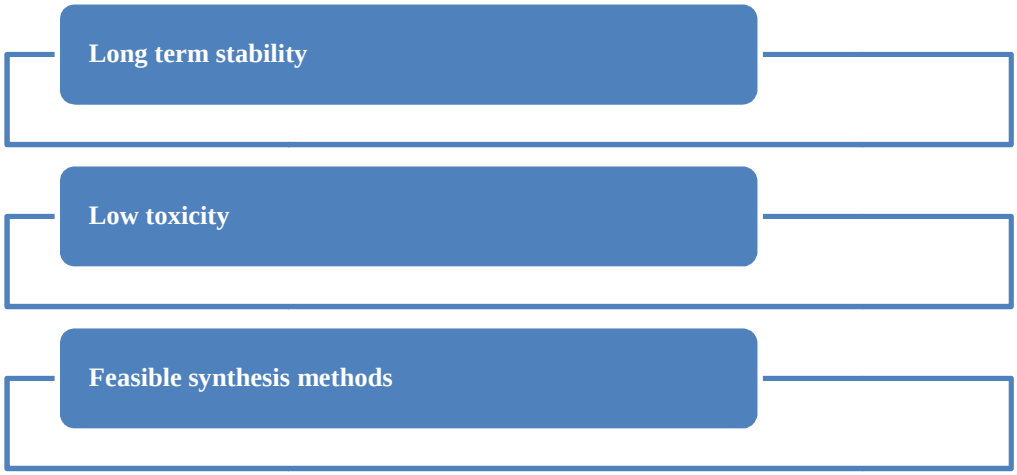

Figure 5: Future Directions of MXenes Research

VII. CONCLUSION

MXenes are a novel 2D material family with numerous applications in sensing and energy. This chapter discussed MXenes' bottom-up and top-down synthesis methodologies, as well as their distinctive features and possible applications. Researchers in environmental science, biomedical engineering, conversion, catalysis, and energy storage are all interested in transition metal carbides and nitrides. Molecular two-dimensional MXene nanoparticles are predicted to lay the groundwork for a new generation of revolutions in chemical science, physical science, and Materials Science in the near future. MXenes have shown outstanding performance in energy storage, sensors, and supercapacitors, indicating that this new class of 2D materials has promising applications in next-generation energy storage and conversion devices.

VIII. ACKNOWLEDGEMENTS

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