2D MXenes as Co-catalysts in Photocatalysis:
Synthetic Methods

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HIGHLIGHTS

- Two-dimensional transition metal carbides/nitrides (MXenes) as co-catalysts were summarized and classified according to the different synthesis methods used: mechanical mixing, self-assembly, in situ decoration, and oxidation.
- The working mechanism for MXenes application in photocatalysis was discussed. The improved photocatalytic performance was attributed to enhancement of charge separation and suppression of charge recombination.

ABSTRACT  Since their seminal discovery in 2011, two-dimensional (2D) transition metal carbides/nitrides known as MXenes, that constitute a large family of 2D materials, have been targeted toward various applications due to their outstanding electronic properties. MXenes functioning as co-catalyst in combination with certain photocatalysts have been applied in photocatalytic systems to enhance photogenerated charge separation, suppress rapid charge recombination, and convert solar energy into chemical energy or use it in the degradation of organic compounds. The photocatalytic performance greatly depends on the composition and morphology of the photocatalyst, which, in turn, are determined by the method of preparation used. Here, we review the four different synthesis methods (mechanical mixing, self-assembly, in situ decoration, and oxidation) reported for MXenes in view of their application as co-catalyst in photocatalysis. In addition, the working mechanism for MXenes application in photocatalysis is discussed and an outlook for future research is also provided.

KEYWORDS  MXenes; Photocatalysis; Co-catalyst; Synthetic methods
1 Introduction

Energy shortage and environmental pollution have become the two major issues faced by humanity due to limited fossil fuel resources and increasing consumption. Developing sustainable and clean energy is the key to addressing these two problems [1–15]. In being clean and inexhaustible, solar energy shows great potential to be one of the most promising future energy sources. Solar energy can be exploited in photovoltaic technologies [16], CO₂ photoreduction [17, 18], N₂ photo-fixation [19], degradation of organic compounds [20–26], and photocatalytic water splitting [27]. In renewable hydrogen fuel-based photocatalytic water-splitting systems [28–30], photocatalysts play a critical role [31, 32]. Photo-catalyzed solar energy conversion can be divided into three steps: (1) light absorption, (2) charge separation and transfer, and (3) surface reaction. Any improvement on each of these steps will contribute to enhancing the total conversion efficiency. Conventional photocatalysts such as TiO₂, g-C3N4, and CdS demonstrate low photocatalytic efficiency due to rapid charge recombination in these materials. Using noble metals such as Pt, Ru, and Pd as co-catalysts will increase cost, although such materials can enhance charge separation ability and suppress recombination of charges. A co-catalyst that is both efficient and cheap is thus urgently needed to promote the development of photocatalysis.

MXenes, comprising transition metal carbides, nitrides, and carbonitrides, are a new family of two-dimensional (2D) materials that have attracted much attention in recent years [2]. The general formula of MXene is M_{n+1}Xₙ (n = 1, 2, 3), where M represents a transition metal, such as Sc, Ti, Zr, Hf, V, Nb, Ta, and Mo, while X represents C and/or N. Owing to their unique structure and superior photoelectronic properties, layered structure MXenes show various potential applications in different areas, such as energy storage [3, 33–38], electromagnetic interference shielding [39, 40], gas sensors [41], wireless communication [42], water treatment [43, 44], solar cells [45–47], and catalysis [41, 48–51]. 2D MXenes are being increasingly studied in the past few years, as evidenced by the rapidly increasing number of scientific articles published per year (Fig. 1a). MXenes are usually synthesized by selectively etching the A layer from MAX phases, which constitute a family of tertiary ductile ceramics, where the A layer is made of an element such as Al, Ga [52], or Si [53]. After selective etching of the A layer, 2D MX layers with surface functional groups (–O, –OH, –F, or a mixture of several groups denoted as Tₓ) are left. The most widely used methods for selective etching are wet chemical HF etching and in situ HF etching (using a mixture of acids and fluoride salts), although other routes using tetramethylammonium hydroxide (TMAOH) [54, 55], electrochemical [56, 57], or etching with NaOH [58], and ZnCl₂ [49] have also been explored. Generally, multilayered MXenes are produced by HF etching, whereas single or few-layered MXene flakes are obtained by in situ HF etching or through delamination of a multilayered MXene by intercalation of large organic molecules (Fig. 1b). The etching methods of Ti₃C₂Tₓ MXene, which is the first discovered and the most studied MXene, have been reviewed elsewhere [59, 60].

In view of the rapid development in the application of 2D MXenes, several reviews on their synthesis [59–61], and application in energy storage [33, 48, 62] and catalysis

![Fig. 1](a) The rapid expansion of 2D MXenes materials and (b) the most widely used methods to synthesize MXenes
have been reported. MXenes are promising for application in photocatalysis because of their large surface area, good conductivity, presence of a sufficient number of active sites, and containing suitable elements for effective photocatalysis, but they cannot be directly used as photocatalysts since MXenes are generally not semiconductors. Although there are some MXene semiconductors that have been predicted theoretically, these have not yet been experimentally synthesized. In this review, we give a detailed discussion on MXene as a co-catalyst in photocatalysis and describe the different methods used for the synthesis of MXene-derived photocatalysts, along with problems encountered in this system and a prospective outlook on future research in this field.

2 Synthetic Methods for MXenes as Co-catalysts in Photocatalysis

In view of their good conductivity and large surface area, MXenes have been applied in photocatalysis both to replace noble metal co-catalysts and to enhance the charge separation ability of the photocatalyst. The most common methods used for the preparation of photocatalyst composites include mechanical mixing, self-assembly, in situ decoration and oxidation, or a combination of the three methods.

2.1 Mechanical Mixing and Self-assembly

Mechanical mixing is the easiest method to form photocatalyst composites. Stirring the two components in the liquid phase or grinding of powders can be used for sample preparation. Interestingly, due to electrostatic attraction, photocatalysts with positive charge are easily combined with MXenes whose surfaces are enriched with negative charges, leading to self-assembled photocatalyst composites. In addition, the self-assembling property could be further improved by using other induced techniques simultaneously, where the photocatalysts and co-catalysts are prepared in advance.

An et al. demonstrated that synergetic effects of Ti$_3$C$_2$ MXene and Pt when used as dual co-catalysts enhanced the photoactivity of g-C$_3$N$_4$ for hydrogen evolution, where HF-etched exfoliated Ti$_3$C$_2$ and g-C$_3$N$_4$ were mixed in liquid by stirring followed by photodeposition of Pt on the composites. The photoactivity of the dual...

![Fig. 2 Schematic showing charge separation between MXene co-catalyst and a photocatalyst taken from Ye et al. Reprinted with permission from Ref. [69]. Copyright 2018 John Wiley & Sons. Ran et al. Reprinted with permission from Ref. [70]. Copyright 2017 Nature Publishing Group. Peng et al. Reprinted with permission from Ref. [71]. Copyright 2016 American Chemical Society](image-url)
co-catalysts-modified photocatalysts (g-C_3N_4/Ti_3C_2/Pt) was much better than that of Pt- or Ti_3C_2-only systems, reaching 5.1 mmol h^{-1} g^{-1} in hydrogen production (Fig. 4a). This enhanced performance was due to the presence of Ti_3C_2 MXene that facilitated interfacial charge separation and carrier transport from the conduction band (CB) of g-C_3N_4 to Pt. Our group prepared g-C_3N_4/Ti_3C_2Tx composites by grinding g-C_3N_4 and Ti_3C_2Tx powders together followed by annealing in different gas atmospheres, to tune the surface termination groups (Fig. 4b) [74]. X-ray photoelectron spectroscopy data showed an increase in –O termination groups accompanied by a decrease in –F termination groups on the surface of Ti_3C_2. Ti_3C_2 with –O termination groups had better photoactivity, revealing that the presence of such groups in Ti_3C_2 had a positive effect on hydrogen production by increasing the number of active sites. Moreover, this finding was consistent with density functional theory (DFT) simulation results. The ΔΔG_H of Ti_3C_2 with –O terminations was found to be as low as 0.01 eV, which is lower than that of Pt (111). In a similar study, Ye et al. [69] treated HF-etched Ti_3C_2 with KOH to convert –F groups into –OH groups, and then combined the KOH-treated Ti_3C_2 with TiO_2 (P25) powder by stirring in water (Fig. 3c). DFT calculations demonstrated that –OH groups played the role of active sites for the adsorption and activation of CO_2 reduction [69]. Experimentally, the photoactivities for CO_2 reduction were increased 3 times and 277 times after KOH treatment, for CO and CH_4, respectively (Fig. 4d). Interestingly, increasing the number of –OH groups not only improved the photo-conversion efficiency but also changed the nature of the products. The –OH groups resulting from KOH treatment provided more active sites for CO_2 adsorption and enabled greater electron transfer to CO_2 and facilitated its reduction to CH_4. Though the surface termination groups can be changed through annealing and KOH treatments, –F groups could not be completely exchanged. More studies to precisely tailor the termination groups need to be carried out in the future.

![Fig. 3 TEM images of photocatalysts combined with a MXene by mechanical mixing taken from a An et al. Reprinted with permission from Ref. [72]. Copyright 2018 The Royal Society of Chemistry. b Xie et al. Reprinted with permission from Ref. [73]. Copyright 2018 Elsevier. c Ye et al. Reprinted with permission from Ref. [69]. Copyright 2018 John Wiley & Sons. d Liu et al. Reprinted with permission from Ref. [44]. Copyright 2018 Elsevier](https://doi.org/10.1007/s40820-019-0309-6)
Xie et al. [73] used an electrostatic self-assembly process to combine positively charged CdS nanosheets and Ti$_3$C$_2$ nanosheets (possessing negative charge) (Fig. 3b) for CO$_2$ reduction (Fig. 4c). Cai et al. [75] synthesized Ag$_3$PO$_4$/Ti$_3$C$_2$ by electrostatically driven self-assembly method, which had the advantage of being a mild method that prevented Ti$_3$C$_2$ from oxidation. The composites showed better performance than reduced graphene oxide (rGO), and this preparation procedure provided a new direction to the preparation of semiconductor-MXene composites. Liu et al. [44] fabricated a 2D layered and stacked g-C$_3$N$_4$/Ti$_3$C$_2$ composite by evaporation-induced self-assembly and used it to degrade...
organic pollutants (ciprofloxacin) (Fig. 3d). Both photogenerated holes and superoxide radicals (\(\cdot O_2^-\)) resulting from ciprofloxacin decomposition (Fig. 4f); in this process, self-assembly was an efficient method that allowed intimate mixing of the components in the composite. The sample was also more homogeneous than mechanically mixed ones because of the electrostatic attraction between the charged entities. However, opposite charges on each surface were required for self-assembly, which limited wider application of this process. Therefore, other techniques to induce self-assembly such as evaporation-induced self-assembly were developed to widen the range of application of products [44].

The above-mentioned MXene-based composites prepared by mechanical mixing and self-assembly methods for photocatalysis application are summarized in Table 1. Results from all these works prove that 2D MXene is an efficient additive material to enhance charge separation and charge transfer during photocatalysis. In these two methods, the properties of MXenes are retained by avoiding high temperature and use of other solvents or surfactant. No change in oxidation or surface termination groups occurs in these synthesis methods. Therefore, these two are the easiest and allow synthesis under the mildest conditions.

2.2 In Situ Decoration of Semiconductors onto the Surface of MXenes

In contrast to composites prepared by mechanical mixing of materials, in situ decoration methods consist in synthesizing a different material directly onto the MXene surface. As a result, in situ synthetized materials and MXenes are chemically bonded, which could be an important advantage in some designs. However, the range of viable synthetic conditions for in situ decoration is limited, because MXenes are easily oxidized in solution, especially at high temperatures [107]. It is therefore necessary to use mild conditions to protect MXenes from oxidation, especially when mono- and few-layered MXenes are used. So far, g-C_3N_4, TiO_2, CdS, and bismuth compounds have been bonded to various MXenes using this strategy.

g-C_3N_4 is one 2D semiconductor material that is combined with MXenes used as a co-catalyst in the photocatalysis process (Fig. 5). MXene can be added during the calcination of a precursor, such as melamine and thiourea, but the high calcination temperature (around 550 °C) may cause the oxidation of MXene into TiO_2. The high photoactivity of g-C_3N_4/MXene is attributed to the efficient charge separation; moreover, the heterojunction formed by TiO_2/g-C_3N_4 also plays an important role in charge separation [108]. Shao et al. [81] synthesized Ti_2C/g-C_3N_4 by melamine calcination and used it in hydrogen production (Fig. 5a, d). Though the ratio of Ti_2C in the composite was as low as 0.4 wt%, a peak due to TiO_2 resulting from the oxidation of Ti_2C could be seen in the XRD pattern. Liu et al. [19] synthesized TiO_2/C/g-C_3N_4 heterojunction by melamine calcination (Fig. 5b), where Ti_3C_2 was oxidized to TiO_2/C during the calcination process. This composite was highly effective in the reaction of nitrogen reduction to ammonia, with the best performance reaching as high as 250.6 μmol h^{-1} g^{-1}, which was better than that of TiO_2/C and g-C_3N_4 (Fig. 5e). Xu et al. [82] synthesized Ti^{3+}-rich Ti_3C_2/g-C_3N_4 by calcination of thiourea and employed it as an electrode for CO_2 reduction in a photoelectrochemical (PEC) system (Fig. 5c, f), achieving a total CO_2 reduction rate of 25.1 mmol h^{-1} g^{-1}. The Ti^{3+} species suppressed charge recombination at the Ti_3C_2/g-C_3N_4 heterojunctions, leading to a corresponding increase in CO_2 conversion efficiency.

Apart from the above-mentioned synthesis methods, composite photocatalysts can also be synthesized by combining TiO_2, a metal sulfide, or a bismuthide with MXene under hydrothermal conditions (Fig. 6). Gao et al. [83] synthesized TiO_2/Ti_3C_2 nanocomposites by a hydrothermal method using TiSO_4 as a precursor for methyl orange (MO) degradation (Fig. 6a), where small TiO_2 particles could be observed on the surface of multilayered Ti_3C_2. Wang et al. [84] employed TiCl_4 as the precursor in the hydrothermal synthesis of rutile TiO_2/Ti_3C_2T_x for hydrogen production by water splitting (Fig. 6d). The photocatalytic activity of TiO_2 when combined with other MXenes (Ti_2CT_x and Nb_5CT_x flakes) as co-catalysts was also explored; results proved that in general, MXenes could be used as effective co-catalysts for solar hydrogen production. Ran et al. [70] combined CdS and Ti_3C_2 particles by a one-step hydrothermal reaction (Fig. 6b). A hydrogen production rate of 14,342 μmol h^{-1} g^{-1} was achieved when using Ti_3C_2 as the co-catalyst; this performance is 136.6 times higher than that of the pure CdS photocatalyst. The effectiveness and versatility of Ti_3C_2 MXene as a co-catalyst for photocatalytic hydrogen production was demonstrated by other metal sulfides (ZnS) [91] photocatalysts as well. Xie et al. [73] showed that Ti_3C_2
### Table 1 MXene-based composites prepared by different synthetic methods for photocatalysis applications

| Sample | MXene (synthetic method) | Sample synthesis | Reactant | Sacrificial agent | Rate | Precursor | Refs. |
|--------|--------------------------|------------------|----------|-------------------|------|-----------|-------|
| g-C3N4/3%Ti3C2/2%Pt | Ti3C2 flakes (HF 48%, 20 h, 60 °C and H2O delamination, 12 h, ultrasonication) | (1) Ti3C2 stirring dispersions (2) Pt UV deposition | H2O | 10 vol% triethanolamine (TEOA) | 5100 μmol/h gcat. | – | An et al. [72] |
| g-C3N4/Ti3C2T (1:0.3) | Multilayer Ti3C2 (HF 49%, 24 h) | Grinding in a mortar | H2O | 10 vol% TEOA | 88 μmol/h gcat. | – | Sun et al. [74] |
| CdS/0.5%Ti3C2T | Ti3C2 flakes (LiF 1 g/HCl 9 M, 24 h, 35 °C) | (1) Ultrasonication (2) Stirring in water | 4-NA | 40 mg ammonium formate in 30 mL solution | 180 mg/L/h | – | Xie et al. [73] |
| P25/5%Ti3C2-OH | Multilayer Ti3C2 (HF 49%, 24 h and KOH 2 M, 4 h) | Stirring in water | CO2 | – | 28.35 μmol/h gcat. | – | Ye et al. [69] |
| a-Fe2O3/Ti3C2 (1:2) | Multilayer Ti3C2 | (1) Stirring in ethanol (2) Ultrasonication | Rhodamine B (RhB) | – | 5 mg/L/h | – | Zhang et al. [76] |
| g-C3N4/Ti3C2 (100:3) | Ti3C2 flakes (HF 40%, 24 h and H2O intercalation, 5 h, ultrasonication) | (1) Ultrasonication (2) Stirring in water at 60 °C | Ciprofloxacin | – | 18 mg/L/h | – | Liu et al. [44] |
| TiO2/5%Ti3C2 | Ti3C2 flakes (LiF 1 g/HCl 6 M, 24 h, 35 °C) | Sonication | H2O | 25% Methanol | 2650 μmol/h gcat. | – | Su et al. [77] |
| Ag3PO4/2%Ti3C2 | Ti3C2 flakes (NaF 3.35 g/HCl 36–38 wt%, 12 h, 60 °C) | (1) Stirring in water with AgNO3 (2) Adding Na2HPO4 | H2O | Tetracycline hydrochloride (TC-H) etc. | – | 192 mg/L/h | – | Cai et al. [75] |
| 3%Ti3C2/g-C3N4 | Ti3C2 flakes (LiF 1.5 g/HCl 16 M, 24 h, 35 °C) | (1) Sonication in HCl (2) Stirring | H2O | 10 vol% TEOA | 73.3 μmol/h gcat. | – | Su et al. [78] |
| TiO2/0.5%Ti3C2/1%CoS | Multilayer Ti3C2 (HF 49%, 4 h) | (1) Stirring in 2-methylimidazole (2) Hydrothermal 140 °C for 12 h with thioacetamide | H2O | 20 vol% methanol | 950 μmol/h gcat. | Co(NO3)2 2-methylimidazole and thioacetamide | Zhao et al. [79] |
| Sample | MXene (synthetic method) | Sample synthesis | Reactant | Sacrificial agent | Rate | Precursor | Refs. |
|--------|--------------------------|------------------|----------|------------------|------|-----------|-------|
| CdS/MoS2/2%Ti3C2Tx | Ti3C2 flakes (HF 49%, 72 h, ultrasonication in H2O, 2 h) | (1) MoS2 synthesis (2) Stirring with Ti3C2 (3) Add CH3N2S and Cd(CH3COO)2 (4) Hydrothermal 160 °C for 24 h | H2O | 0.25 M Na2S and 0.35 M Na2SO3 | 9679 μmol/h/gcat. | Cd(CH3COO)2, CH3N2S, MoS2 | Chen et al. [80] |
| 0.4%Ti2C/g-C3N4 | Ti2C flakes (NH4F 16 g/HCl 9 M, 24 h) | (1) Stirring ethanol | H2O | 10 vol% TEOA | 950 μmol/h/gcat. | Melamine | Shao et al. [81] |
| 10%TiO2/C/g-C3N4 | Multilayer Ti3C2 (HF 49%, 4 h) | (1) Stirring in water (2) 550 °C, 2 h in muffle | N2 | 20 vol% methanol | 250 μmol/h/gcat. | Melamine | Liu et al. [19] |
| Pd-Ti3C2/g-C3N4 (1:10) | Multilayer Ti3C2 (HF 40%, 24 h) | (1) Grinding (2) 500 °C, 2 h in muffle (3) Pd electrodeposition | CO2 | 0.1 M KHCO3 | 25,100 μmol/h/gcat. | Thiourea | Xu et al. [82] |
| 0.001 molTiO2/Ti3C2 | Multilayer Ti3C2 (HF 49%, 24 h, 60 °C) | (1) Stirring (2) Hydrothermal 180 °C, 18 h | Methyl orange (MO) | – | 40 mg/L/h | TiSO4 | Gao et al. [83] |
| TiO2/5%Ti3C2 | Ti3C2 flakes (HF 48%, 15 h and DMSO delamination, 15 h) | (1) Stirring in ice-water bath (2) Heated 95 °C, 4 h | H2O | 25% methanol | 43 μmol/h/gcat. | TiCl4 | Wang et al. [84] |
| TiO2/5%TiC | TiC flakes (HF 10%, 10 h and DMSO delamination) | | | | | | |
| TiO2/5%Nb2C | Nb2C flakes (HF 48%, 90 h and 20% isopropyl alcohol delamination) | | | | | | |
| CdS/2.5%Ti3C2 | Ti3C2 nanoparticles (HF 49%, 20 h, 60 °C and H2O delamination, ultrasonication, 5 h) | (1) Stirring in water (2) Hydrothermal 180 °C, 12 h | H2O | Lactic acid (88 vol%) | 14,342 μmol/h/g | Cd(Ac)2 | Ran et al. [70] |
| Sample | MXene (synthetic method) | Sample synthesis | Reactant | Sacrificial agent | Rate | Precursor | Refs. |
|--------|--------------------------|------------------|----------|------------------|------|-----------|-------|
| TiO₂/C/BiVO₄ (1:1079) | Ti₃C₂ flakes (LiF 1.5 g/HCl 16 M, 48 h, 50 °C) | (1) Stirring in water (2) Hydrothermal 100 °C, 6 h | RhB | – | 3.1 mg/L/h | Bi(NO₃)₃ | Shi et al. [85] |
| TiO₂/Ti₃C₂ (1:1) | Multilayer Ti₃C₂ (HF 40%, 26 h, 60 °C) | (1) Stirring in 10 M NaOH (2) Hydrothermal 180 °C, 10 h | Methylene blue (MB) | – | 8.5 mg/L/h | NH₄VO₃ | P25 | Luo et al. [86] |
| BiOBr/Ti₃C₂ (250:1) | Ti₃C₂ flakes (LiF 3 g/HCl 9 M, 24 h, 35 °C) | (1) Stirring (2) Refluxed 80 °C, 2 h | RhB | – | 24 mg/L/h | Bi(NO₃)₃ and KBr | Liu et al. [87] |
| 2%Ti₃C₂/Bi₂WO₆ | Ti₃C₂ flakes (HF 40%, 72 h and DMSO delamination, ultrasonication, 1 h) | (1) Stirring (2) Hydrothermal 120 °C, 24 h | CO₂ | – | 2.22 μmol/h/gcat. | Bi(NO₃)₃ | Cao et al. [88] |
| Bi₀.⁹Gd₀.₁Fe₀.⁸Sn₀.₂O₃/Ti₃C₂ | Multilayer Ti₃C₂ (HF 39%, 36 h) | (1) Stirring in 0.01 M acetic acid and ethylene glycol (2) Sonicated, 2 h, 60 °C (3) stirring 1 h, 80 °C | Congo red | – | – | Na₂WO₄ | Bi₁₋ₓGdₓFe₁₋ₓSnₓ | Tariq et al. [89] |
| In₂S₃/TiO₂@Ti₃C₂ | Multilayer Ti₃C₂ (HF 50%, 20 h) | (1) Stirring (2) Hydrothermal 180 °C, 24 h | MO | – | 18 mg/L/h | In(NO₃)₃ | Wang et al. [90] |
| ZnS/0.75 wt%Ti₃C₂ | Ti₃C₂ flakes (HF, 24 h, 25 °C) | (1) Stirring in ethanol–glycerol (2) Hydrothermal 180 °C, 10 h | H₂O | 20 vol% lactic acid | 502.6 μmol/h/gcat. | CH₃CSNH₂ | ZnCl₂ | Tie et al. [91] |
| Ti₃C/3%TiO₂/1%Ag | Multilayer Ti₂C (HF 48%) | (1) Stirring for volatiles evaporation (2) Annealing in H₂ at 400 °C | Salicylic acid | – | 32.4 μmol/h | Titanium isoproplate | Wojciechowski et al. [92] |
Table 1 (continued)

| Sample | MXene (synthetic method) | Sample synthesis | Reactant | Sacrificial agent | Rate | Precursor | Refs. |
|--------|--------------------------|------------------|----------|------------------|------|-----------|-------|
| TiO2/Ti3C2 (12 h) | Multilayer Ti3C2 (HF 49%, 12 h, 60 °C) | Hydrothermal 160 °C for different time, NaBF4 and HCl | MO | – | 24 mg/L/h | – | Peng et al. [71] |
| TiO2/Ti3C2 (20 h) | Multilayer Ti3C2 (HF 49%, 12 h, 60 °C) | Hydrothermal 200 °C for different time, NH4F | MB | – | 6 mg/L/h | – | Peng et al. [93] |
| HC-TiO2 | Ti3C2 flakes (tetramethylammonium hydroxide 25%, 24 h) | Hydrothermal 160 °C, 9 h | H2O | 10 vol% TEOA | 33.04 μmol/h/gcat. | – | Jia et al. [94] |
| 4%Cu/TiO2@Ti3C2_T_x 12 h | Multilayer Ti3C2 (HF 49%, 12 h, 60 °C) | (1) Hydrothermal 160 °C for different time, NaBF4 and HCl (2) Photodepositing copper nanodots | H2O | 6.7 vol% methanol | 764 μmol/h/gcat. | – | Peng et al. [95] |
| Ti3C2/TiO2/CuO (100:1) | Multilayer Ti3C2 (HF 49%, 24 h, 60 °C) | (1) Dissolved in water (2) Annealing in argon, 500 °C, 30 min | MO | – | 15 mg/L/h | – | Lu et al. [96] |
| C/TiO2 700 °C-150 sccm | Multilayer Ti3C2 (HF 40%, 90 h, 55 °C) | Heated in CO2 at different temperature and different rate, 1 h | H2O | 10 vol% TEOA | 480 μmol/h/gcat. | – | Yuan et al. [97] |
| TiO2/Ti3C2 (TT550 °C) | Multilayer Ti3C2 (HF 50%, 48 h) | Calcination at different temperature | CO2 | – | 4.4 μmol/h/gcat. | – | Low et al. [98] |
| Nb2O5/C/Nb2C-1 h | Multilayer NbC (HF 50%, 90 h) | Annealing in CO2, 850 °C for different time | H2O | 25% methanol | 7.81 μmol/h/gcat. | – | Su et al. [99] |
| Microporous-MXene/TiO2 nanodots | Multilayer Ti3C2 (HF 50%, 90 h) | High-energy ball milling in air, 1.5 h, 200 rpm | RhB, etc. | – | – | – | Cheng et al. [100] |
| C/TiO2 | Multilayer Ti3C2 (HF 40%, 2.5 h) | | MB | – | 2.13 mg/L/h | – | Li et al. [101] |
| Sample | MXene (synthetic method) | Sample synthesis | Reactant | Sacrificial agent | Rate | Precursor | Refs. |
|--------|--------------------------|------------------|---------|------------------|------|-----------|-------|
| TiO₂/Ti₃C₂@AC-48 h | Multilayer Ti₃C₂ (HF 49%, 24 h) | Heated in H₂O for different time at 60 °C | H₂O | 29 g/L ascorbic acid (AA) | 33.4 μmol/h/gcat. | – | Sun et al. [102] |
| Ti₃C₂/TiO₂-500/Pt | Multilayer Ti₃C₂ (HF 40%, 72 h) | (1) Hydrothermal in 1 M NaOH and 30% H₂O₂, 140 °C, 12 h (2) Immersed in 0.1 M HCl, 24 h (3) Annealing in muffle for different time | H₂O | 20 vol% methanol | H₂ 1596.35 μmol/h/gcat. | – | Li et al. [103] |
| LDC-S-TiO₂/C | Multilayer Ti₃C₂ (HF 40%, 48 h, 45 °C) | (1) Ball mixing with sulfur (2) Hydrothermal 155 °C, 12 h (3) Annealing in CO₂ at 700 °C for 2 h (4) Annealing in air at 450 °C, 2 h | H₂O | 0.01 M AgNO₃ | O₂ 500 μmol/h/gcat. | – | Yuan et al. [104] |
| TiO₂/Ti₃C₂ | Multilayer Ti₃C₂ (HF 30%, 10 h, 40 °C) | Hydrothermal 160 °C for 12 h, NaBF₄ and HCl | Carbamazepine | – | 1.48 mg/L/h | – | Shahzad et al. [105] |
| Ti₃C₂/TiO₂/15%MoS₂ | Multilayer Ti₃C₂ (HF 40%, 72 h) | (1) Hydrothermal 160 °C for 12 h with NaBF₄ and HCl (2) Hydrothermal 200 °C for 24 h with Na₂MoO₄ and CN₂H₃S | H₂O | TEOA | 6425 μmol/h/gcat. | NaBF₄, HCl, Na₂MoO₄ and CN₂H₃S | Li et al. [106] |
flakes enabled the local confinement of Cd²⁺ released during photo-corrosion and thus enhanced the stability of the metal sulfide. Besides CdS, In₂S₃/Ti₃C₂Tx hybrids synthesized by hydrothermal method have been used for methyl orange degradation as reported by Wang et al. [90]. Among the hybrids based on other additives (carbon nanotubes (CNT), rGO, MoS₂, and TiO₂), Ti₃C₂-based composites showed the best photocatalytic activity, which is attributed to their high electrical conductivity. Shi et al. [85] synthesized TiO₂/C/BiVO₄ composites by hydrothermal method for the degradation of Rhodamine B, where Ti₃C₂ was employed both as a support for the growth of BiVO₄ nanoparticles and as a precursor for the generation of 2D-carbon upon oxidation. The electron transfer process was accelerated by the presence of Ti₃C₂-derived 2D-carbon layers, thus improving the photocatalytic performance for Rhodamine B degradation. Ultrathin 2D/2D heterojunction of MXene/Bi₂WO₆ prepared by the in situ growth of ultrathin Bi₂WO₆ nanosheets on the surface of ultrathin Ti₃C₂ nanosheets for photocatalytic CO₂ reduction was reported by Cao et al. [88] (Fig. 6c). The CH₃ and CH₂OH yield were 4.6 times higher than those obtained with pristine Bi₂WO₆, which was ascribed to the enhanced CO₂ adsorption arising from the increased specific surface area and improved pore structure of the layered heterojunction. The different composites/hybrids containing MXene or MXene-derived products prepared by hydrothermal methods and used in photocatalysis are listed in Table 1.

The synthetic process for MXenes-based composites includes doping into the photocatalysts or using MXene as a support for in situ decoration of the semiconductor photocatalyst. The chemical reactions taking place during photocatalyst formation led to increased interfacial area, thus providing greater possibilities for the transfer of photogenerated electrons. However, one disadvantage of this method is the oxidation of MXenes during photocatalyst synthesis. Although difficult to precisely characterize, conditions of formation of the photocatalysts may be too harsh and cause structural degradation of MXenes, especially in the case of single-layered MXenes, due to their lower stability toward oxidation.
2.3 MXene-Derived Photocatalysts

Different from mechanical mixing, self-assembly, and decoration methods, the in situ oxidation method using MXene (Ti$_3$C$_2$ is the most studied example) as a precursor for the synthesis of photocatalysts has also been explored (Fig. 7). Peng’s group tuned the facet of TiO$_2$/Ti$_3$C$_2$ using a hydrothermal method without using an additional TiO$_2$ precursor (Fig. 7a, b) [71, 93]. NaBF$_4$ and NH$_4$F were used as reagents to, respectively, control morphology in the synthesis of (001) TiO$_2$/Ti$_3$C$_2$ and (111) TiO$_2$/Ti$_3$C$_2$, which were then applied in methyl orange degradation. Both the facet type of TiO$_2$ and the ratio of TiO$_2$ to Ti$_3$C$_2$ could be controlled by changing the duration of the hydrothermal reaction. Jia et al. [94] obtained closely aggregated TiO$_2$ nanorods with high carbon doping starting from Ti$_3$C$_2$ flakes and demonstrated a better photoactivity than commercially available P25 for hydrogen production (Fig. 7c). The carbon doping also changed the electron structure of TiO$_2$ and enhanced its light absorption ability. Peng et al. [95] also used Ti$_3$C$_2$ as a hole trap and Cu as an electron trap to separate the charges through a dual-carrier-separation mechanism, showing the potential of MXene as an efficient functional material for photocatalysis (Fig. 7d).

Calcination under atmosphere containing gases such as CO$_2$ and O$_2$ is another method used for the controlled oxidation of MXenes (Fig. 8). Lu et al. [96] obtained Ti$_3$C$_2$/TiO$_2$/CuO by annealing Cu(NO$_3$)$_2$ and Ti$_3$C$_2$ together under argon atmosphere (Fig. 8a). Because of its good electronic conductivity, the incorporation of Ti$_3$C$_2$ improved electron/hole separation and led to better methyl orange degradation. Yuan et al. [97] annealed Ti$_3$C$_2$ in CO$_2$ to prepare 2D-layered C/TiO$_2$ hybrids used in hydrogen production, in which the presence of 2D carbon layers increased electron transport channels and enhanced charge separation efficiency (Fig. 8b). In addition, the effects of oxidation temperature
and CO₂ on the grain size and crystal structure of TiO₂ were also investigated, revealing that increasing oxidation temperature and CO₂ gas flux led to larger grain sizes and more rutile TiO₂ formation. Low et al. [98] calcined Ti₃C₂ at different temperatures, enabling the in situ growth of TiO₂ nanoparticles on Ti₃C₂ nanosheets, thus forming TiO₂/Ti₃C₂ composites with different loading amounts of TiO₂ with the aim to improve performance in CO₂ reduction reaction (Fig. 8c). Interestingly, three main products were obtained during the photocatalytic CO₂ reduction process due to the sufficiently high intrinsic reduction potential of TiO₂. Results of the study also pointed out that excess of Ti₃C₂ in the composite could have an adverse effect on photocatalytic performance. Su et al. [99] used CO₂ to partially oxidize Nb₂C to form Nb₂O₅/Nb₂C composites for hydrogen production, where Nb₂O₅ and metallic Nb₂C served, respectively, as the semiconductor photocatalyst and co-catalyst (Fig. 8d). The easily formed junction at the interface served as an electron sink to efficiently capture photogenerated electrons and suppress recombination of photogenerated electron–hole pairs, thus enhancing the efficiency of charge separation and contributing to improved photocatalytic activity [71, 93, 99, 102].

Besides the hydrothermal method and calcination, other routes such as chemical oxidation and high-energy ball milling were also used to oxidize MXenes (Fig. 9). Cheng et al. [100] oxidized Ti₃C₂ flakes with 30% H₂O₂ to form microporous-MXene/TiO₂₋ₓ nanodots (Fig. 9a). This composite worked as a photo-Fenton bifunctional catalyst for Rhodamine B degradation under both dark and illumination conditions. Li et al. [101] synthesized TiO₂@C nanosheets from Ti₂C by high-energy ball milling and used it for methylene blue degradation (Fig. 9b). Shortly thereafter, our group used water to oxidize Ti₃C₂ to be applied in hydrogen
production using Eosin Y as a sensitizer [102]. Similar to other oxidized MXenes, amorphous carbon and TiO₂ were formed after oxidation (Fig. 9c, d). The various MXene-derived composites obtained by in-situ oxidation to be used as photocatalysts are listed in Table 1.

The MXenes oxidation is different from other methods because of the residual presence of carbon (mostly amorphous carbon) after oxidation, and the M element is oxidized into metal oxide on the carbon layer. Thus, the composite obtained is of the form metal oxide/MXenes/C. Both MXenes and C can be used as co-catalysts in the photocatalysis process. However, in this method, the ratio of the photocatalyst to MXenes varies within a certain range since no precursor is introduced. The limitation of this method is that only a few semiconductors (depending on M element) can be used as the photocatalyst.

3 Mechanism of MXenes as Co-catalysts

Since MXenes are conductors and serve as co-catalysts, the mechanism of action of a MXenes-based photocatalytic system is through accelerated charge separation and suppression of carrier recombination [69–71]. The photocatalysts absorb visible light and photogenerated electrons are excited to the CB, while holes are left in the valence band (VB). The excited charge carriers are transferred to MXenes at the interface mainly because of the higher potential of MXenes. Electrons transfer to MXenes without recombination and react on the MXene surface to generate H₂ by reducing H⁺ [74, 78, 81, 91, 94, 102, 103], CH₄ and CO by reducing CO₂ [88, 98], or NH₃ by reducing N₂ [19], as shown in Fig. 10 process (a). In process (b), holes transfer to MXenes and react to produce OH⁻ that can be utilized for degradation of organics [71, 93, 95]; electrons can also produce OH⁻ for organic degradation [71, 93]. The charge transfer process
from the photocatalyst to MXenes improves electron–hole pair separation and suppresses charge recombination in photocatalysts, thus enhancing the photoactivity.

Another advantage of using MXenes in photocatalysis is due to their termination groups. For example, −O termination groups show the best potential for hydrogen production because of their low $|\Delta G|_{H}$ and the availability of active sites for the adsorption of hydrogen atoms [70, 74]. Though termination groups are important in photocatalysis, currently, it has not been possible to precisely control the relative concentrations of the different termination groups. Using presently available synthetic methods, changing the different reaction conditions can partially modify the termination groups on MXenes surface and thereby affect their performance in photocatalysis.

4 Conclusion and Outlook

In summary, the application of MXenes in photocatalysis has shown rapid development since 2015. Among the MXenes family, Ti$_3$C$_2$ has been the most studied MXene. Mechanical mixing and self-assembly are mild and easy methods of synthesis, where the ratio of MXenes to the photocatalyst can be controlled. In addition, MXenes can also be doped into the photocatalysts by in situ decoration of a semiconductor photocatalyst. The large interfacial area afforded by
the doping process improves electron transfer. However, the MXenes oxidation method has the advantage of obtaining both carbon and MXenes as co-catalysts by forming a metal oxide/MXenes/C structure. Though the above-mentioned four synthetic methods are generally used for photocatalysts, with further development in the field of MXenes, new processes may be discovered.

Besides developing improved synthetic methods, the other aspects that need to be focused on in the future are as follows:

1. Controlling the morphologies of MXenes. MXene flakes show larger surface area than multilayered MXenes, since mono- or few-layered MXenes provide a greater number of active sites for photocatalytic reactions. The flakes are also convenient for building structures, such as quantum dots, spheres, and nanorods. However, the instability of MXenes should be taken into account during heat treatment [107].

2. MXenes combine with efficient photocatalysts. MXenes can be used as co-catalysts to combine with many semiconductor photocatalysts due to their excellent electronic conductivity and the presence of numerous hydrophilic groups on the surface. Hundreds of semiconductor photocatalysts have been reported for photocatalysis so far. Attention should be paid to combining the efficient and cheap photocatalysts with MXenes to achieve better photocatalytic performance. So far, only g-C₃N₄, CdS, ZnS, TiO₂, CuO, Nb₂O₅, BiVO₄, Ag₃PO₄, α-Fe₂O₃, In₂S₃, Bi₂WO₆, BiₓMo₆Oₓ₋ₓ, Fe₇S₁₉O₄, and BiOBr have been explored, with TiO₂ and g-C₃N₄ attracting the most attention.

3. Surface modification of MXenes. Surface termination groups significantly affect the properties of MXenes, and thus, tuning the surface termination groups and modifying the MXenes surface are expected to greatly influence its potential as co-catalyst.

4. Synthesis of new MXenes. To date, only a small fraction of the different possible MXenes has been synthesized in laboratories. Some MXenes showing semiconducting properties have been reported based on theoretical calculations. Theoretical predictions help in the synthesis of semiconductor MXenes and applied in photocatalysis. Once obtained experimentally, potential MXenes can be applied as photocatalysts, thus widening the application range of MXenes. Moreover, new types of transition metal borides (MBenes) have also been predicted [34, 109] and have shown potential for photocatalysis applications. More work needs to be done in this direction.

5. Developing new synthesis methods for MXenes. HF and in situ HF wet chemical treatment are by far the most used methods in MXenes synthesis. Other HF-free methods are emerging and leading to MXenes with different properties. Yet, these have not been investigated in photocatalytic applications, and thus, the effect of the type of synthesis process used on the final performance of the MXene is currently not understood.

In short, due to tremendous effort of scientists worldwide, the great potential of MXenes in photocatalysis has been revealed. With the fast-growing development in this area, it is expected that more and more studies will focus on the applications of MXenes photocatalysis and pave the way to the commercialization of photocatalytic technologies based on these materials.

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