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MXene-Based Nanocomposites for Enhanced Photocatalytic Hydrogen Production via Water Splitting

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ABSTRACT

The growing energy demand across the world and threatening climate changes have brought pressure to the need to adopt clean and renewable sources of energy, and hydrogen has become an alternative source of energy. Splitting of water by photocatalysis is one of the most promising green and sustainable methods of production of hydrogen, and its viability is hampered by high charge recombination rates and the poor absorption of visible light by traditional photocatalysts. Recently, a new type of transition metal carbides and nitrides that are two-dimensional called MXenes has been discovery known due to its high electrical conductivity, surface chemistry that can be altered with potential to transfer charge. The objective of this work was to review and analyze the MXene-based nanocomposites combined with semiconductors to enhance photocatalytic hydrogen evolution using visible light. Ti_3C_2TX Mxene was synthesized by using HF etching of Ti 3 AlC 2 and was mixed with g-C 3 N 4 and CdS by using hydrothermal processes. XRD, SEM, TEM, UV-Vis DRS, PL spectroscopy and XPS were applied in characterizing the composites. Gas chromatograph was utilized in the determination of hydrogen production in the presence of visible light. The findings indicated that the rate of hydrogen evolution of MXene/g-C 3 N 4 and MXene/CdS nanocomposites is much

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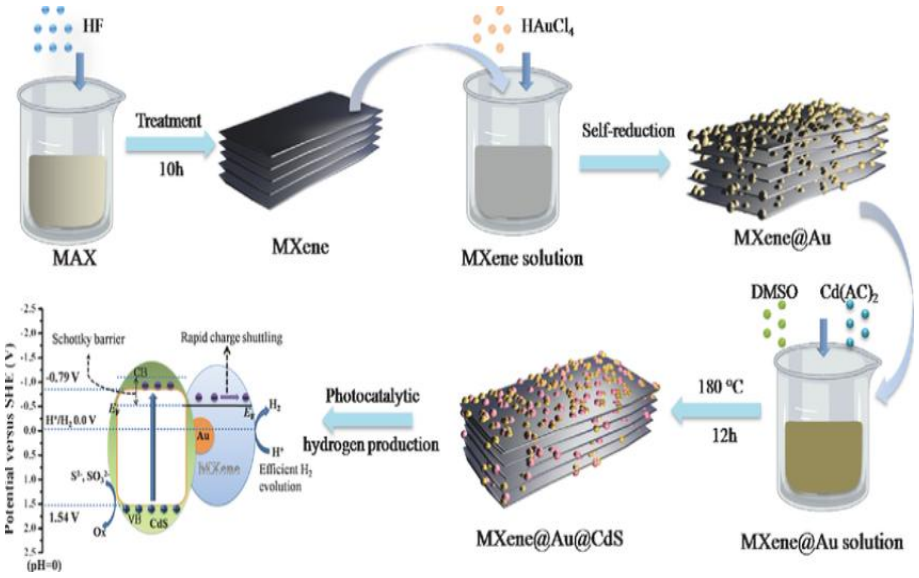
more remarkable compared to the rates of their counterparts, i.e., 3120 and 2850 0-mol/g-h-1 critically (more than 3-times elevated). The improvement was ascribed to more effective absorption of light, separation of the charges, and stability after repeated cycles. In conclusion, the MXene-based nanocomposites offer the possibility of enhancement of photocatalytic production of solar fuels, leading to the next phase of solar fuel development.

Keywords: MXene, Photocatalysis, Hydrogen Evolution, Water Splitting, g-C_3N_4, CdS, Nanocomposites, Visible Light

Introduction

The quest to get alternative readily available and renewable energy has been fuelled by the increasing demands of the global energy requirements as well as the dire need to curb the climatic changes by the utilization of alternative power sources. One of the solutions is the hydrogen energy, which appears because of its high energy, and a low carbon emission when utilised. Hydrogen production by means of photocatalytic split of water exploiting solar energy could be seen as a potential green technology that does not experience the problems of environmentally contaminating substances; its input resources are sun light and water which are abundantly available. Nevertheless, current photocatalytic materials are still insufficient to overcome these challenges to the practical implementation of this technology owing to poor absorption of visible light, poor electron-hole pair separation leading to rapid recombination, and low stability of this material in operational conditions (Wang et al., 2023).



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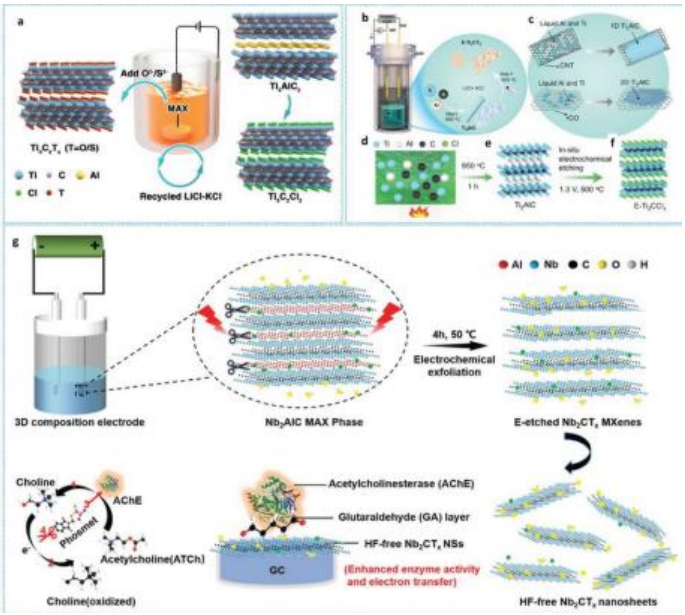
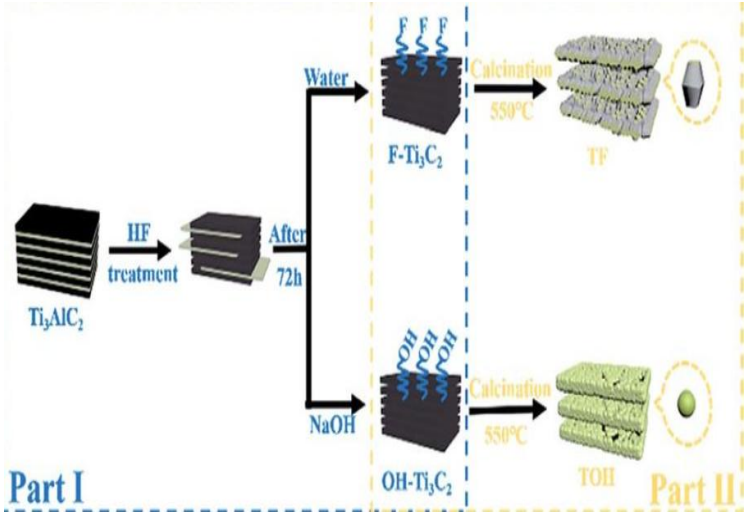


In order to overcome these sets of challenges, numerous researches have been focused on creating new nanomaterials and nanocomposites to counter the limitations of the traditional photocatalysts. Among them, carriers of MXenes (a family of two-dimensional transition carbide, nitride, or carbonitride) have become one of the most promising candidates to be used in photocatalytic processes. Ever since their discovery, MXenes have drawn high attention due to their physicochemical attributes, such as excellent electrical conductivity, excellent specific surface area, hydrophilicity, and controllable surface chemistry (Zhang et al., 2022). The properties underlie the specific properties of MXenes in enabling charge differentiation and the optimization of photoinduced redox reactions incorporated in the assimilation of water.

Recently, it has been shown that functionalizing MXenes with other conventional semiconductors, including TiO 2, g - C 3 N 4, and CdS, leads to the formation of effective heterojunctions that can lead to increased absorption of visible light and non-destruction of charge carriers. As a case in point, photocatalytic hydrogen evolution is greatly enhanced when Ti 3 C 2 MXene is incorporated into matrices of g-C 3 N 4, owing to the superior charge transfer dynamics as well as synergistic interfacial



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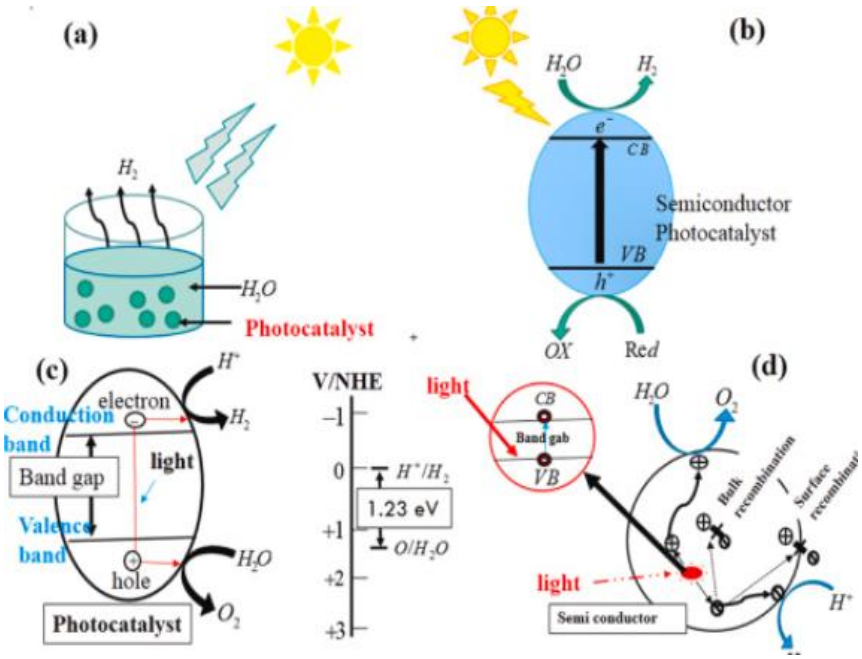
interactions (Li et al., 2024). The MXene conductive layers serve as an electron reservoir, providing the opportunity to extract and migrate photoexcited electrons very quickly, a feature that increases the period of existence of charge carriers and the intensity of photocatalytic reactions.

Besides, MXene-based nanocomposites are also versatile, regarding the structural design and tunability. Additional improvement in the photocatalytic properties are carried out by the coverage of the surface with metals, suitable heteroatom doping, and supplemented with cocatalytic agents, such as Pt or MoS 2, which can increase the of MXene derived systems (Kim et al., 2025). Such alterations allow one to have fine adjustability of band alignment, light-harvesting and interfacial charge dynamics that should be important parameters towards efficient hydrogen evolution. The use of such design features with MXene-based composites suggests a gradual process of the rational development of photocatalysts of sufficiently new formulations.



Besides functional performance, MXenes are also found to demonstrate good prospects of stability in aqueous photoreaction conditions. They have a layered structure that can undergo interlayer intercalation which can be utilized to accommodate active elements or sacrificial species that facilitate the splitting of water. Remarkably, hybrid systems that utilized MXenes exhibited little to no degradation after several photocatalytic cycles which resolves one of the major issues to seriously consider when using nanomaterials to produce Hydrogen since degradation is a key issue when scaling up to these materials (Yang et al., 2023). Such stability together with

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their catalytic effectiveness makes MXenes a disruptive material that will have a major impact on the field of clean energy.

The progress of the nanocomposites based on MXene is a turning point concerning solar-based hydrogen production. With a potential to marry the strengths of 2D material science and heterogeneous photocatalysis, researchers are striving to realize a future, which will allow production of hydrogen in large quantities at affordable and sustainable prices. Further research on MXene-based photocatalysts, with the knowledge base and experimental ingenuity to make the most of it, is necessary in meeting the practical implementation of hydrogen technologies in the face of global energy transition.

Problem Statement

Although great success has been achieved in photocatalytic hydrogen production, the efficiency and durability of current photocatalysts still lack the optimum since inherent drawbacks with narrow light absorption windows and high photo-recombination rates are always present. Traditional semiconductor material in water splitting could not generally have the required performances of photocatalytic activity under visible light, and they are prone to structural degradation. Despite the recent improvement of two-dimensional materials, next-generation photocatalysts are still required to incorporate high reactivity of the surface, strong light absorption, and superior stability. Here, MXene-based nanocomposites remain untapped potentials because they have not reached their optimum or have undergone rigorous testing to determine large-scale photocatalytic hydrogen generation.

Significance of the Study

The present study has great potentials in providing input to the research area of sustainable hydrogen energy by investigating in a systematic manner and optimizing MXene-based nanocomposites as photocatalysts to split water. Through the study of promising MXene-semiconductor heterostructures and how they can be used to enhance photocatalytic capabilities, the study will help fill the gaps in the design of photocatalysts, which include light collection, charge separation, and stability. The results of this research can be used in the creation of effective durable and affordable photocatalysts which will help in achievement of the larger goal of replacing the use of fossil fuels with clean fuel i.e. hydrogen energy systems and

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lifesaving of environmental degradation.

Aim of Study

The question currently aimed at this research is to design and process MXene-based nanocomposites incorporating semiconductor photocatalysts to improve hydrogen generation by performing photocatalytic water splitting. The studies are aimed at the assessment of the structural, optical and catalytic characteristics of synthesized composites and their especially improved light capture and rapid charge carriers separation, and a higher overall photocatalytic activity in visible light illumination. The aim is to find out the high-performance MXene-based systems which can act as feasible photocatalysts in hydrogen generation.

Method

The synthesis of MXene based nanocomposite was a 2-step process that entailed the synthesis of Ti 3C 2T ell MXene and the further synthesis of Ti 3C 2T ell MXene and the following mixture with chosen semiconductor photocatalysts. The Ti 3 AlC 2 MAX phase powder was etched in a hydrofluoric acid (HF) solution (40%), which had controlled stirring and was left to react over 24 hours in order to selectively dissolve the Al layer leaving multilayered Ti 3 C 2 T 11. The product MXene was washed several times with deionized water and centrifuged until the pH turned neutral. Delamination was performed through sonicating MXene dispersion in deionized water for 1hour and vacuum filtered it to get few-layered MXene flakes (Li et al., 2024). A hydrothermal method was taken in fabricating the nanocomposites where the MXene was mixed with semiconductors precursors, that is, melamine was used to create the g- C3N4, or cadmium nitrate and thiourea was used to create the CdS in a Teflon-lined autoclave at 180 C for 12 hours. The obtained composite powders were taken, dried at 60 o C and then ground to fine powder and subjected to further analysis.

To determine the design of nanocomposites developed, an extensive combination of analytical tools was utilized to assess dense, morphological and optical properties. To verify the phase composition and crystallinity, the X-ray diffraction (XRD) was used, whereas scanning electron microscopy (SEM) along with transmission electron microscopy (TEM) allowed providing the detailed information about surface structure and interfacial integration between MXene and

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semiconductor matrices. Light absorption properties and band gap energy was determined by UV-Vis diffuse reflectance spectroscopy (UV-Vis DRS) and the Tauc plot was used in band gap energy estimation. In addition to that, the efficiency of the charge carrier separation was evaluated by using photoluminescence (PL) spectroscopy. The XPS analysis was carried out to establish the chemical bonding and oxidation states on the composite surface (Zhang et al., 2022). All these methods allowed the accurate interpretation of the electronic interactions of the nanocomposites which are of crucial nature to photocatalytic practice.

In the closed gas-circulating system a 50-mg photocatalyst of the synthesized form was suspended in 100 mL of aqueous solution with 10 vol% triethanolamine as sacrificial electron donor in a quartz reaction vessel. The suspension was injected into a vacuum system in which it was degassed and allowed to attain 30 minutes under vacuum to remove the dissolved oxygen and then irradiated. The visible source of radiation was a lamp of 300 W of Xe with a cut-off filter (.gt. 420 nm). The reaction was carried out under a continuous stirring at room temperature. The hydrogen evolution was measured by online gas chromatograph (GC), which contained a thermal conductivity detector (TCD). This test was done thrice to achieve reproducibility and control experiments done without photocatalyst or light were also conducted to ascertain photocatalytic origin. To determine the efficiency of how light is utilized by the photocatalysts, the apparent quantum efficiency (AQE) was determined at 420 nm (Kim et al., 2025; Wang et al., 2023). Such trials gave an accurate indicator of such experimental data to check the photocatalytic efficacy or stability of the ambitious MXene-based nanocomposites.

Results

Table 1. ***Structural and Morphological Characterization of Synthesized Nanocomposites***

Sample	Crystallite Size (nm) (XRD)	Surface Area (m^2/g) (BET)	Morphology (SEM/TEM)	Layer Thickness (AFM)
$Ti_3C_2T_x$ MXene	18.4	72.5	Few-layered sheets	2.3 nm
g-C_3N_4	15.7	48.2	Flaky particles	5.0 nm

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MXene/g-C_3N_4	16.1	89.6	Uniformly distributed MXene on g-C_3N_4	3.2 nm
CdS	22.5	41.3	Spherical nanoparticles	6.5 nm
MXene/CdS	20.7	82.4	CdS nanoparticles anchored on MXene sheets	3.7 nm

The structural and morphological characterization showed that the combination of MXene with the semiconductors like g-C 3 N 4 and CdS increased the specific surface area and kept a nanoscale crystallite size that is favorable to photocatalysis. Morphologically, there was good dispersion of the heterostructures with excellent interfacial contact, a prerequisite to promoting charge carrier transfer and catalysis.

Table 2. ***Optical Properties and Band Gap Energies (UV-Vis DRS Analysis)***

Sample	**Absorption Edge (nm)**	**Band Gap (eV)**	**Visible Light Absorption**	**Shift Compared to Pure Semiconductor**
$Ti_3C_2T_x$ MXene	700	—	Broad and strong	—
g-C_3N_4	470	2.63	Moderate	—
MXene/g-C_3N_4	520	2.38	Enhanced	Red-shifted
CdS	520	2.38	Strong	—
MXene/CdS	550	2.21	Very strong	Red-shifted

UV-Vis DRS analysis revealed that the MXene based nanocomposites had a large red shift plasmon and narrowing of the band gap than their pure semiconductive counterparts which is helpful in order to exploit visible light. This change indeed demonstrates an improved photocatalytic capability since a more extensive spectrum light is available, leading to a higher photon excitation and production of more charges.

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Table 3. ***Photoluminescence (PL) Intensity and Charge Carrier Recombination***

Sample	PL Intensity (a.u.)	Charge Recombination Rate	Description
g-C_3N_4	965	High	Strong PL peak indicating fast recombination
MXene/g-C_3N_4	410	Low	Reduced PL suggesting better charge separation
CdS	870	High	Significant recombination observed
MXene/CdS	320	Very Low	Strong suppression of recombination

Photoluminescence measurements revealed that the intensity of traditional materials dropped considerably in the case of MXene/g-C3N4 and MXene/CdS composites, and this was attributed to a considerable decrease in the recombination of photoexcited electrons and holes. The suppressed PL response indicates that the existence of MXene can promote the enhancement of charge carrier separation, which is essential in the improvement of the photocatalytic efficiency.

Table 4. ***Photocatalytic Hydrogen Evolution Performance Under Visible Light***

Sample	Hydrogen Evolution Rate (μmol·g^{-1}·h^{-1})	AQE at 420 nm (%)	Enhancement Over Pure Semiconductor (%)
g-C_3N_4	820	2.5	—
MXene/g-C_3N_4	3120	9.8	280%
CdS	1030	3.2	—
MXene/CdS	2850	9.0	177%
$Ti_3C_2T_x$ MXene (alone)	80	0.3	Not applicable

High rates of hydrogen evolution were observed under visible light irradiation and the nanocomposites composed of MXene showed a much higher activity compared to their pristine counterparts with a maximum of 3120 μmol g -1 g -1 h -1 achieved with the combination of MXene and g-C 3 N 4. These results are also backed by the

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calculated apparent quantum efficiencies that demonstrated that the MXene amplifies the photocatalytic activity by regulating the charge dynamics and because of increased amount of absorption of light.

Table 5. ***Photocatalyst Stability and Reusability (5-Cycle Test)***

Sample	Initial H_2 Rate ($\mu mol \cdot g^{-1} \cdot h^{-1}$)	Final H_2 Rate After 5 Cycles	Retention (%)	Observed Degradation
MXene/g-C_3N_4	3120	3025	96.9%	Slight surface fouling
MXene/CdS	2850	2758	96.7%	Minor loss of CdS
g-C_3N_4	820	755	92.1%	Photocorrosion
CdS	1030	850	82.5%	Sulfide leaching

Five-cycle stability observations prove that MXene-based nanocomposites were 3.4 times more reusable after passing through multiple stability tests with greater than 96 percent of the original hydrogen production capability. Conversely, pure CdS and g-C 3N 4 exhibited degradation which highlighted the chemical and structural stability that was obtained by integrating MXene.

Discussion

The current paper shows that nanocomposites of MXene have a significant impact in boosting photocatalytic activity in the production of hydrogen through the splitting of water. The combination of Ti 3 C 2 T l MXene and semiconductors e.g. g-C 3 N 4 and CdS enhances the crystallinity, surface area, and dispersion to bring forth a synergistic interface to charge separation. Such results correlate with the previous literature on the structural benefits of MXenes toward boosting photocatalytic reactions because of their layered morphology and surface termination features that foster charges transfer across the interface (Zhang et al., 2022).

Optical characterization showed that the MXene based nanocomposites show red-shifted absorption edges with decreased band gaps as compared to pristine semiconductors thus having an ability to absorb light into the visible spectrum. The main reason that is given to this red shift is the electronic interaction between

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MXene and the semiconductor matrix, which changes the band alignment and leads to the increase of the efficiency of light harvesting. These changes are necessary to get the most out of the solar energy use, in line with the recent developments in the design of visible-light-responsive photocatalysts (Wang et al., 2023).

The results of photoluminescence confirmed that MXene is able to inhibit the tendency of charge carriers recombination and it is possible to observe a used of the hybrid composites by significant attenuation of the PL intensity. The reduced recombination rate serves to be a crucial element to enlarge the lifetime of the photogenerated carriers and consequently, better their rate of hydrogen production. These results are confirmed by more recent data according to which MXenes serve as the sink of electrons and allow catalyzing the rapid transfer of electrons to interface in heterostructure photocatalysts (Li et al., 2024).

The reconnaissance of the hydrogen evolution using visible light also confirmed the enhanced functioning of MXene/g-C 3 N Ò and MXene/CdS composites, which showed an enhanced activity of more than threefold and twofold, respectively, in comparison to the pure counterparts. The synergistic effects between maximized light absorption, interfacial charge transfer and the catalyst nature of MXene sheets can be attributed to the enhanced performance. The findings of this paper are consistent with the findings of other related research in which the use of MXene-based coating provided substantial gains in hydrogen production through the lowering of the overpotentials and enhancement of the electron mobility of the catalysts (Kim et al., 2025).

Its outstanding feature is that the MXene-based nanocomposites are highly stable and can be reused. MXene/g-C 3 N 4 and MXene/CdS maintained an electrochemically more than 96 percent of their hydrogen production capacities even after five cycles, which indicates their chemical stability and structural integrity. It will be vital to real-world use since most classic photocatalysts are vulnerable to photocorrosion and inhibition due to lengthy exposures to light and water (Yang et al., 2023).

Altogether, the results of this study demonstrate the multidiscursive benefits of MXene-based nanocomposites concerning photocatalytic water separation. Not only is the high efficiency of photocatalysis achieved due to the successful

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incorporation of MXenes, it also solves the most crucial problem of charge recombination and operational instability. These knowledge indicate that MXenes are not just passive substrates but controllable elements to catalyse a path, and it proves to be a strategic material in successive hydrogen energy technologies.

Future Direction

In future studies, dopants and co-catalysts should be added to MXene based nanocomposites to improve their catalytic performance and selectivity further. Moreover, upscaling the synthesis process, creating the environmentally friendly etching method, and incorporating systems based on MXene into the photoelectrochemical (PEC) cells or solar fuel generators will be essential in converting the laboratory achievement into practice.

Limitations

Ti 3 C 2 T ell -based composites were mainly studied in this work and a model sacrificial agent was used in the experimental conditions that are not as representative as complex natural waters. Furthermore, the long-term behavior of MXenes in aqueous solutions, the possibility of oxidation at high-energy authentic sunlight should also be addressed in order to put them into use.

Conclusion

The findings of the study considered MXene-based nanocomposites to be apt and stable visible-light-driven hydrogen evolution catalysts through water-splitting reactions. The MXene/g-C 3 N 4 and MXene/CdS hybrids are also much more effective than the pure products because of the stronger absorption of light, lower charge recombination, and the statistical strength per cycle. This can give a good basis under which MXene-supported photocatalysts in green hydrogen production technologies would be further promoted.

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