



Application of $Ti_3C_2(OH)_2$ MXene Nanosheets as a Potential Adsorbent and Photocatalyst for Degradation of Organic Dye in Aqueous Media

Batool Hasani Khaneghahi¹ | Shiva Dehghan Abkenar^{1✉} | Javad Gilnezhad² | Mohammad Reza Ganjali² | Morteza Hosseini³

1. Department of Chemistry, Savadkooh Branch, Islamic Azad University, Savadkooh, Iran

2. Center of Excellence in Electrochemistry, School of Chemistry, College of Science, University of Tehran, Tehran, Iran

3. Department of Life Science Engineering, Faculty of New Sciences and Technologies, University of Tehran, Tehran, Iran

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ABSTRACT

In this work, single layered $Ti_3C_2(OH)_2$ MXene nanosheets have been successfully prepared through a facile approach by etching Ti_3AlC_2 with alkaline solution treatment (KOH with minimum amounts of water). The structure and morphology of the produced nanosheets were evaluated through X-ray diffraction (XRD) and field emission scanning electron microscopy (FESEM) analysis and the chemical composition was determined using an energy dispersion X-ray (EDX) spectrometer. Methylene Blue (MB) as a target pollutant adsorption and photocatalytic degradation tests were subsequently performed to assess the functionalities of hydroxyl-terminated MXene. MB removal using $Ti_3C_2(OH)_2$ MXene in the dark in 20 minutes achieved an absorption-desorption balance of 51.2%, and then MB was degraded within 80 minutes under UV light irradiation with great efficiency. Our results presented that the powder of as produced exhibited good photocatalytic activity for three cycles photodegradation. The first-order rate constant (k) was calculated to be 0.0372 1/min. About 97% degradation of Methylene Blue dye in the solution was confirmed within 80 min of exposure to ultraviolet light.

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INTRODUCTION

Water contamination is a common problem in various countries. As most of the water is unusable for agricultural or drinking purposes because of impurities and high salinity levels, only a mere percentage of the earth's water is fresh and appropriate for drinking. Considering the limited fresh water resources, and also the exponential population growth which brings about industrial and agricultural development, it is highly necessary to conserve and protect the water resources (Tiwari et al., 2003). Methylene Blue (MB) and other cationic dyes are a class of organic compounds with ample scientific and industrial applications including the dyeing of cotton, wool and other textiles; coloring of paper; as a hair dyeing agent; as an indicator of

*Corresponding Author Email: dehghan54@yahoo.com

redox reaction in outer space, etc. The major problem with MB is that it is a highly carcinogenic thiazine pollutant of environmental resources (Kumar et al., 2014, Dehghan Abkenar et al., 2019; Tamara and Nidaa, 2019; Geravand et al., 2021).

As synthetic dyes are manufactured as stable and non-degradable molecules, conventional purification approaches are inefficient for their removal from water-based samples. Over the past few years, various technologies such as photocatalytic degradation (Munyai et al., 2021; Pebdeni et al., 2021), sono-catalytic degradation (Pang et al., 2022), biodegradation (Wu et al., 2022), reductive decolorization (Xie et al., 2020) and adsorption (Abkenar et al., 2015; Hajiaghababaei et al., 2017, Dehghan Abkenar et al., 2019) have been used for the elimination of such organic pollutants from water samples.

Recently, a new class of 2D transition metal carbide/nitride materials referred to as MXene have been the focus of attraction for many theoretical and experimental research projects (Naguib et al., 2012; Tran et al., 2021, Qu et al., 2022). Typically, MXene are synthesized by selectively etching A layers of their corresponding source materials, the MAX phases ($M_{n+1}AX_n$), where M and A stand for early transition metal (Sc, Ti, V, Cr, Nb, Mo, Hf) and a group IIIA or IVA elements (Al, Si), respectively, whereas X can be C and/or N. The typical MAX phase materials have the value of $n = 1, 2, \text{ or } 3$ (Khazaei et al., 2013; Ghidiu et al., 2014; Naguib et al., 2014, Anasori et al., 2015). Thus, the $M_{n+1}X_nT_x$ formulation can be considered for MXene, in which M and X are equivalent to that in the MAX phases, and T_x depicts surface termination groups ($-OH, -F, -O$) which are formed throughout the etching process (Khazaei et al., 2017).

Up to now, wet-chemical etching in hydrofluoric acid (HF) or HF-containing or HF-forming etchants has been dominantly used for MXene synthesis, which result in $-O, -F,$ or $-OH$ functional groups as the T_x in $M_{n+1}X_nT_x$. Due to strong chemical bonds between A and M elements in the MAX phase, mechanical exfoliation is practically impossible, thus etching is required (Alhabeib et al., 2017).

The extraction of the Al layer in the Ti_3AlC_2 structure can be done using treatment with KOH in the presence of slight amount of water resulting in total delamination. The etching process led to the replacement of Al atoms with OH groups so a simple washing process can lead to very thin 2D $Ti_3C_2(OH)_2$ nanosheets with a theoretical thickness of 0.95 nm. Studies have shown that the performance of dye absorption by MXene is significantly improved by increasing the number of functional groups ($-OH$) on the surface of nanosheets, due to the reaction in alkaline environment (Wei et al., 2018).

In this study, the nanosheets $Ti_3C_2(OH)_2$ MXene was prepared through a facile approach by KOH solution according to previous literature (Li et al., 2017) and then characterized to corroborate their layered structure. We thus attempted to use mostly hydroxyl (OH)-terminated $Ti_3C_2T_x$ MXene as a potential adsorbent and photocatalyst. Methylene Blue (MB) was selected as a major contamination target since it is an important water contaminant. Therefore, we chose to investigate the interaction of $Ti_3C_2(OH)_2$ MXene with the aqueous dye Methylene Blue (MB) in the dark and under ultraviolet (UV) light. Various factors affecting the removal of the contaminant were carefully monitored and investigated. The dye degradation efficiency (%) and rate constant (k) of catalyst were also measured in order to assess the photocatalytic activity of synthesized MXene.

MATERIALS AND METHODS

pH readings were performed using a Metrohm 713 pH-meter, and absorption spectra measurements were performed with UV-vis Perkin-Elmer lambda 25 spectrometer ranging from 200 to 800 nm by xenon arc lamp. The elemental analysis (Energy-Dispersive X-ray Spectroscopy (EDS)) and the morphology of the nanocomposite were evaluated through map analysis using the TESCAN MIRA3 model of the field emission scanning electron microscope

(FESEM) to obtain relative atomic ratios in the nanocomposite. The crystalline structures of synthetic samples were investigated by X-ray diffraction (XRD, PHILIPS, PW1730) with Cu-K α radiation ($\lambda = 1.54056 \text{ \AA}$).

All the reagents for synthesis and analysis photocatalyst activity were of analytical grade, commercially available and used without further treatments. The glassware employed for experimenting were soaked in diluted nitric acid for about 24 hours prior to experimenting, and then washed repeatedly using DDW. Aluminium powder (60 μm , 99.9% purity), Titanium powder (<45 μm , 99.98% purity) and graphite powder (<45 μm , $\geq 99.99\%$ trace metals basis), were purchased from Sigma-Aldrich(Germany). The MB dye powder with molecular weight 319.85 g/mol, Molecular Formula C₁₆H₁₈ClN₃S and Color Index Number: 52015, was purchased from Sigma Aldrich (Germany). The appropriate of dye was dissolved in DDW in order to attain the stock MB solution(1000 mg/L). The desired concentrations were prepared through diluting the original stock solution. Fig. 1 shows the molecular structure and UV-visible absorption spectrum of standard solutions of MB dye.

For synthesis of Ti₃AlC₂, first Ti, Al and graphite powders with a stoichiometric ratio of 3:1:2 were mixed together in a total of 10 g and placed in a steel ball mill with 450 rpm for 18 h. Then, about 8 g of the mixture was placed at a temperature of 1000 °C for 3 h in a tube furnace under the influence of argon gas flow.

The Ti₃C₂(OH)₂ MXene was prepared by a one-step procedure based on the report of published article (Li et al., 2017). First, the mixture of potassium hydroxide (7 g), Ti₃AlC₂ (1 g), with a small amount of water (1 mL) were lightly ground into paste and thus autoclaved in a polytetrafluoroethylene-lined stainless steel autoclave at 160 °C for 24 h. Subsequently, the solid product was gathered from the autoclave and repeatedly washed with deionized water until the filtrate had a pH of 7. Using a vacuum oven at 70 °C for 12 h the resulting sample was dried out.

MB was used as the target pollutant to investigate the adsorption and photodegradation of powder Ti₃C₂(OH)₂ MXene under visible light irradiation. 20 mg of as-synthesized Ti₃C₂(OH)₂ MXene and different concentration of MB in deionized water were utilized for visible irradiation photocatalytic experiments. In order to reach the adsorption-desorption equilibrium state, the photocatalysts were dispersed in 20 ml of MB solution in the dark at room temperature under continuous stirring for 20 min. A 150 W Xenon Arc Lamp was used for UV light irradiation. The light source was positioned 15 cm above the surface of the reaction solution. Then the aqueous suspensions were stirred under UV light irradiation for various time periods between 5 and 80 min. After the light exposure time, about 5 mL of the mixture was withdrawn and

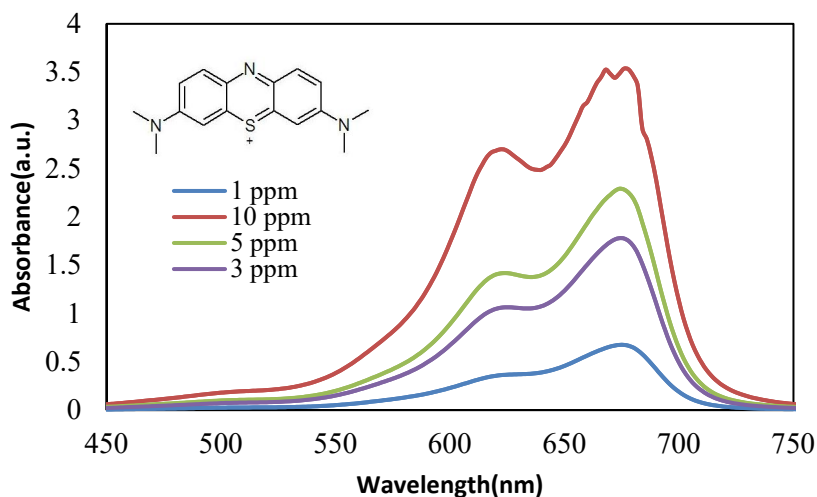


Fig. 1. Molecular structure and UV-visible absorption spectrum of standard solutions of MB dye

centrifugation was done to separate the catalysts prior to the assessment of the photodegradation. Spectrophotometry at 665 nm was employed to measure the MB concentration before and after the experiments. The dye degradation efficiency was calculated using the following Eq(1) (Qu et al., 2022).

$$\%R = \left[\frac{C_o - C_t}{C_o} \right] \times 100 \quad (1)$$

In which C_o and C_t show the initial MB concentration and the concentration at the end of the experiment at time (t) (mgL^{-1}).

We also further evaluated the effects of pH, the amount of the nanosheets $\text{Ti}_3\text{C}_2(\text{OH})_2$ MXene, contact time and irradiation time on the photodegradation efficiency.

RESULTS AND DISCUSSION

The MAX phase and MXene crystallography, microstructure and elemental composition were characterized using XRD, FESEM and EDX correspondingly. The Ti_3AlC_2 structure consists of separate Ti_3C_2 sheets that are connected with a pure Al layer. The extraction of aluminum atoms from the Ti_3AlC_2 structure can break the metal bond between adjacent Ti_3C_2 layers and lead to the production of layered Ti_3C_2 nanosheets. The X-ray diffraction pattern (XRD) of the MXene and MAX are presented in Fig. 2. XRD pattern of MAX phase also showed clear peaks, the strongest of which can be seen at $\sim 9.15^\circ$, $\sim 37.55^\circ$, $\sim 42.7^\circ$, $\sim 60.05^\circ$, $\sim 69.25^\circ$ and $\sim 75.1^\circ$ respectively (Fig. 2a), which are consistent with the previously reported values (Albukhari et al., 2022). The XRD spectrum of MXene after washing and drying exhibited two strong diffraction peaks at $\sim 9.2^\circ$ and $\sim 40.65^\circ$, and the diffraction peak around 38° corresponding to MAX disappeared which indicates the formation of $\text{Ti}_3\text{C}_2(\text{OH})_2$ MXene (Fig. 2b). This results is in accordance with the simulated XRD diffraction peaks of layered structured $\text{Ti}_3\text{C}_2(\text{OH})_2$ reported in previous study (Li et al., 2017).

The morphology evolution of as-prepared $\text{Ti}_3\text{C}_2\text{T}_x$ MXene nanosheets and Ti_3AlC_2 powders are shown by FESEM images. The shape of the particles created from the Ti_3AlC_2 precursor was cracked, and randomly stacked nanosheets combined with some scattered nanosheets in the image. Fig. 3, comparison of images reveals that Ti_3AlC_2 powders have compact, layered morphology (Fig. 3a, b) and the nanosheets of MXene are formed and separated from each other (Fig. 3c, d).

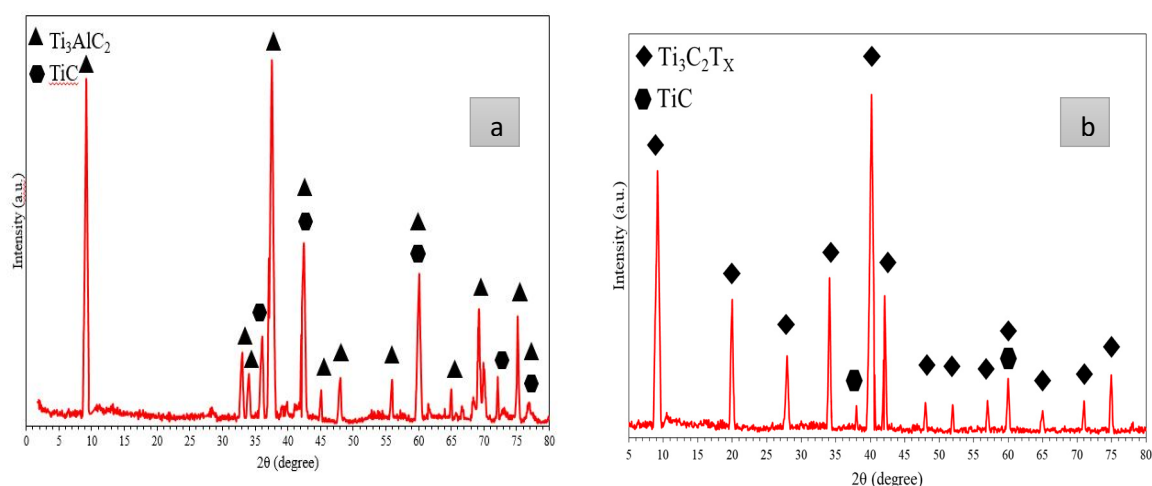


Fig. 2. XRD patterns of (a) MAX phase (b) synthesized MXene nanosheets

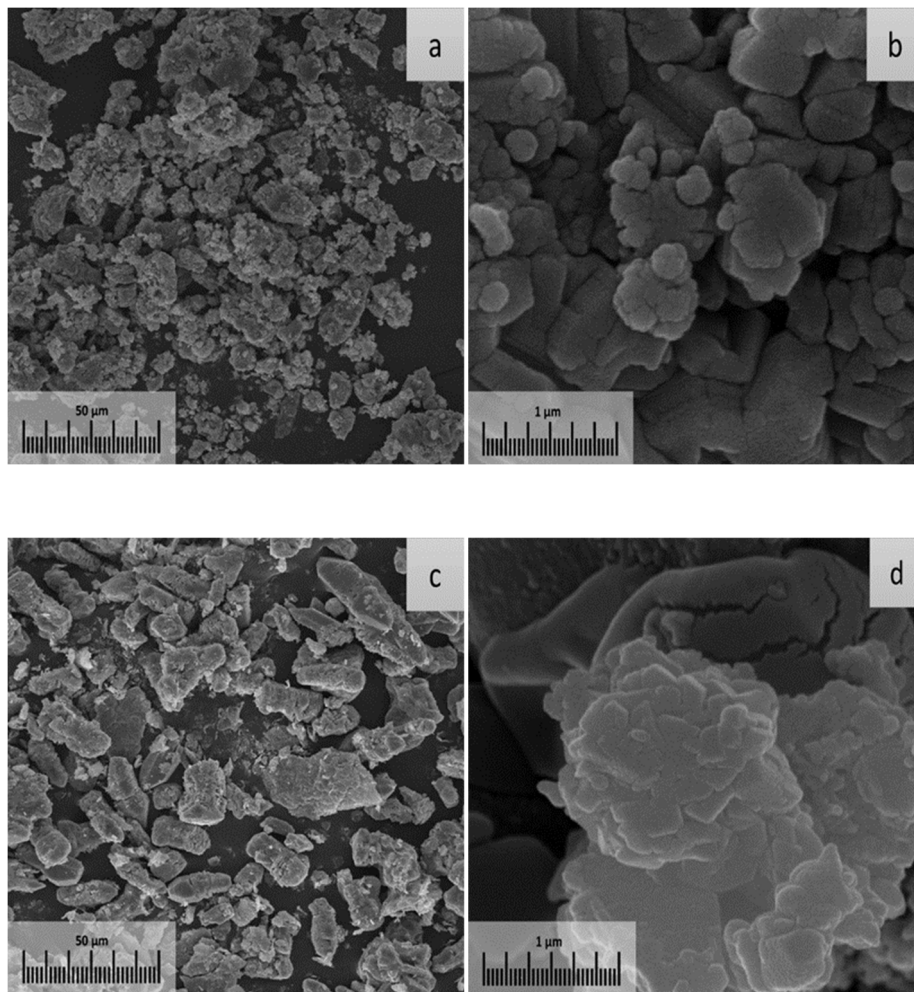


Fig. 3. FESEM of (a), (b) MAX and (c), (d) MXene

The EDX test was used to assess the presence of various elements in the Ti_3AlC_2 and synthesized $Ti_3C_2(OH)_2$ MXene nanosheets, the results of which are shown in Fig. 4. Peaks corresponding to Ti and Al elements were observed in both spectra (Fig 4b, c). According to the results of EDX analysis, a large amount of aluminum has been removed in MAX phase by alkaline etching. As can be seen in Fig 4c, the peak related to Al element has a lower intensity in the spectrum of the synthesized MXene nanosheets, corroborating the partial removal of the Al element from the previous structure and the synthesis of MXene nanosheets. This finding was previously reported in the synthesis of MXene nanosheets (Geravand et al., 2021).

Prior to the dye degradation studies, the adsorption tests were performed under dark conditions. Initially, the adsorption capacity of $Ti_3C_2(OH)_2$ MXene was determined by performing MB adsorption experiments in the dark. A certain amount of MB was dissolved in deionized water to prepare an original 10 mg/L MB solution which was used for all the following experiments. 20 mg of $Ti_3C_2(OH)_2$ MXene was added and dispersed in 20 mL of the as prepared MB solution and subsequently stirred at room temperature in the dark. The UV-Vis absorption spectra of MB in the presence of $Ti_3C_2(OH)_2$ MXene in the dark has been displayed in Fig. 5. As evident, in the first 20 min, most of MB was absorbed and after that point, an adsorption equilibrium was reached. As the adsorption time increased from 20 to 60 minutes, the amount of MB adsorbed increased only a little (Fig. 5), which indicates that 20 minutes can

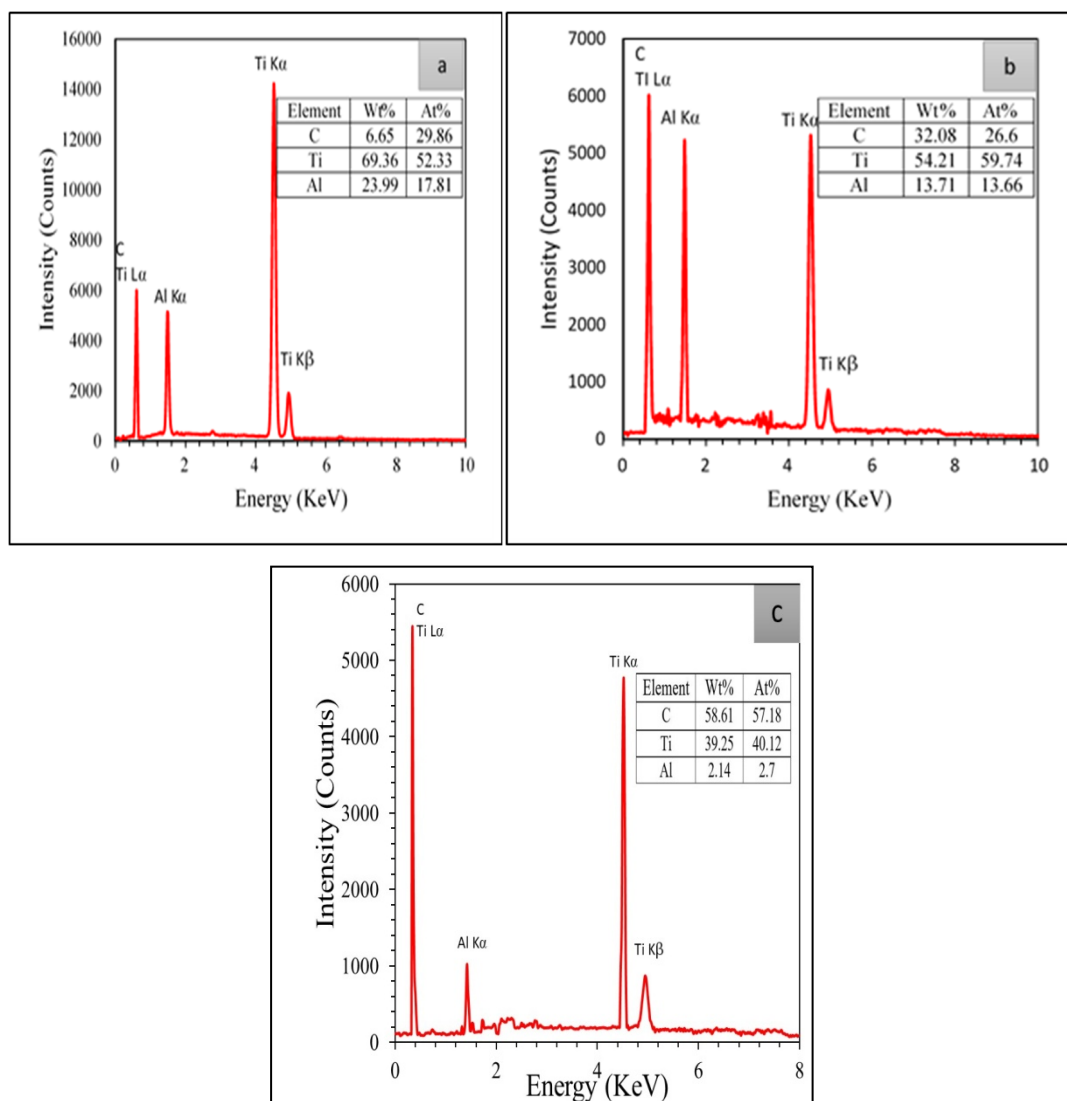


Fig. 4. EDX of mixture of (a) Ti, Al and graphite before ball milling (b) MAX phase (c) synthesized MXene nanosheets

be a suitable adsorption time. Therefore, in subsequent studies on MB degradation, 20 min was set for the adsorption process in the dark to reach adsorption equilibrium before photocatalytic degradation by MXene.

Subsequently, the photocatalytic activities of MXene nanosheets ($\text{Ti}_3\text{C}_2(\text{OH})_2$) were assessed for the degradation of MB solution under ultraviolet light. For this purpose, 4 to 20 mg of photocatalyst were added to the MB solution (10–200 mg/mL) to prepare the reaction suspension. In the dark, the as-prepared aqueous suspensions were stirred for 20 min to establish adsorption–desorption equilibrium. Then stirring was followed under ultraviolet light irradiation for 80 min. After that, about 5 mL of the mixture was taken and centrifuged to separate the catalysts before measuring the photodegradation. UV-Vis spectrophotometry at a wavelength of 665 nm was performed to determine MB concentration. Fig. 6 shows the MB removal using nanomaterials after 80 min of ultraviolet-light irradiation. These results indicate that $\text{Ti}_3\text{C}_2(\text{OH})_2$ MXene nanosheets exhibit great photocatalytic degradation performance.

The pH of the dye solution plays a critical role in the process of adsorption and photodegradation. The pH adjustments were induced using HCl and NaOH solutions and the

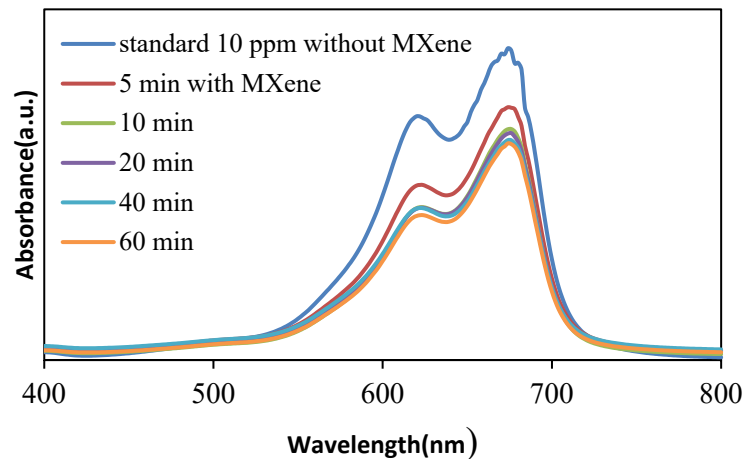


Fig. 5. UV-visible absorption spectra of MB in the presence of $\text{Ti}_3\text{C}_2(\text{OH})_2$ MXene in the dark for adsorption of MB after 5-60 min in dark

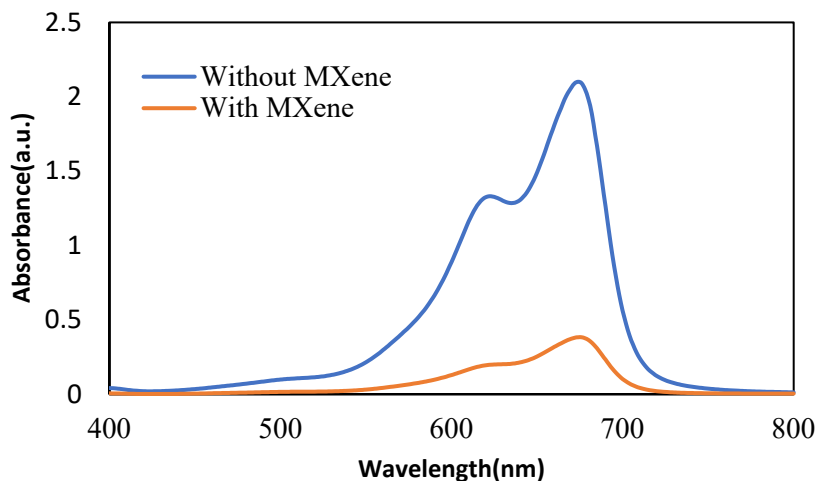


Fig. 6. Photodegradation of MB by $\text{Ti}_3\text{C}_2(\text{OH})_2$ MXene after 80 min ultraviolet-light irradiation

measurements were performed using a digital pH meter. The pH can influence both the aqueous chemistry and surface binding sites of the adsorbent (Pebdeni et al., 2021). In order to find the best pH, the mentioned studies were performed in the pH range of 2.0 to 9.0 with fixed initial concentration and contact time onto an exactly weighed amount of adsorbent (20 mg). The results are given in Fig. 7. It is known that MB exists in cationic form in aqueous media, so it adsorbs to negatively charged surfaces, which can be affected by the presence of hydroxyl groups on the surface of the catalyst. In this work, the treatment of Ti_3AlC_2 with KOH in the presence of a small amounts of water can extract the Al layer. The Al atoms are replaced by OH groups during the etching process, so it is expected that a good electrostatic attraction between the dye and the catalyst can be established. The degradation percentages of MB showed continuous enhancement as the solution pH raised from 2 to 7. The added surface negative charge, which subsequently leads to a strong electrostatic attraction between the MB molecules with positive charge and the functional groups of the used composite, which bear negative charge, can explain this phenomenon. Thus, the pH of the dye solution was set to 7 to complete the study as a moderate value.

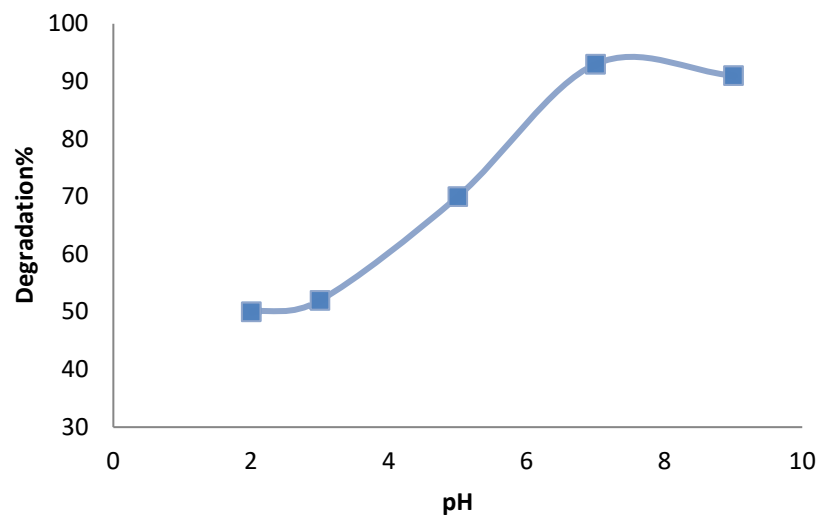


Fig. 7. Effect of pH on the photodegradation of the MB dye

The optimization of the catalyst amount is essential in order to maintain a maximum level of treatment efficiency. MXene amounts ranging from 4 to 20 mg were assessed for the degradation of 10 mL of 20 mg/L MB solution at room temperature and at pH=7 in order to investigate the effect of the catalyst amounts on the photocatalytic reaction under UV light. The corresponding results are depicted in Fig. 8. The obtained results show a significant increase in the percentage of MB degradation with increasing catalyst dosage due to a promising increase in surface area, exposed catalytic active sites and in catalyst adsorption capacity (Abukhadra et al., 2018). It was seen that after 80 min and by employing 20 mg of photocatalyst, the degradation percentage reached 96%.

Various MB concentrations in the range of 10–200 mg/L in the presence 20 mg catalyst after 80 min of light irradiation were assessed in order to investigate the effect of the initial MB concentration on the degradation efficiency. This experiment showed that the initial concentration of the dye is a dominant factor in its reduction percentage in aqueous solution. The results demonstrate that with the increase of the initial concentration of MB,

the amount of dye degradation increases and decreases at a high concentration (200 mg/L). The absorption of dyes on the MXene surface occurs due to the interaction of the substance molecule with the functional groups on the MXene surface. At first, with the increase in dye concentration, the concentration gradient increases and also the possibility of dye collision with absorbent particles increases. Thus, until the active centers of the absorbent are not saturated, the amount of dye removal increases, but at high concentrations, the amount of dye degradation decreases.

We further studied the impact of contact time on degradation using an initial MB concentration of 50 mg/L and 20 mg of adsorbent. Contact time of 5, 20, 30, 40, 60 and 80 min under UV irradiation was investigated on the photocatalytic activity of the MXene on MB degradation, and the results are displayed in fig. 9. A fast decrease in the absorption peak of MB dye was observed as the reaction time increased to 30 min, after which the increase in degradation happened at a slower rate. This behavior shows how free activated sites on fresh catalysts impacts the degradation. As seen in Fig. 9, the reaction rate decreased as the activated sites became saturated.

In order to assess the reaction kinetics of photocatalytic degradation using $\text{Ti}_3\text{C}_2(\text{OH})_2$, the pseudo-first-order kinetic model was applied. Using the Langmuir-Hinshelwood model (Eq. (2)), the rate constants were measured (Pebdeni et al., 2021).

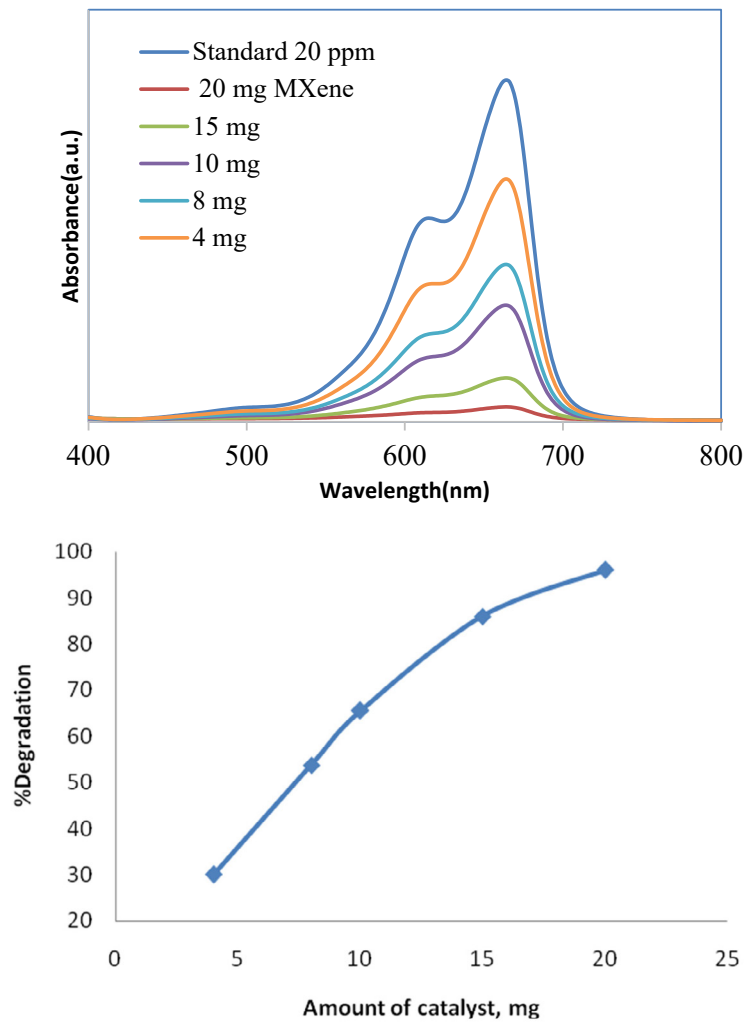


Fig. 8. the effect of the amount of MXene on the photocatalysis reaction under Ultraviolet light irradiation

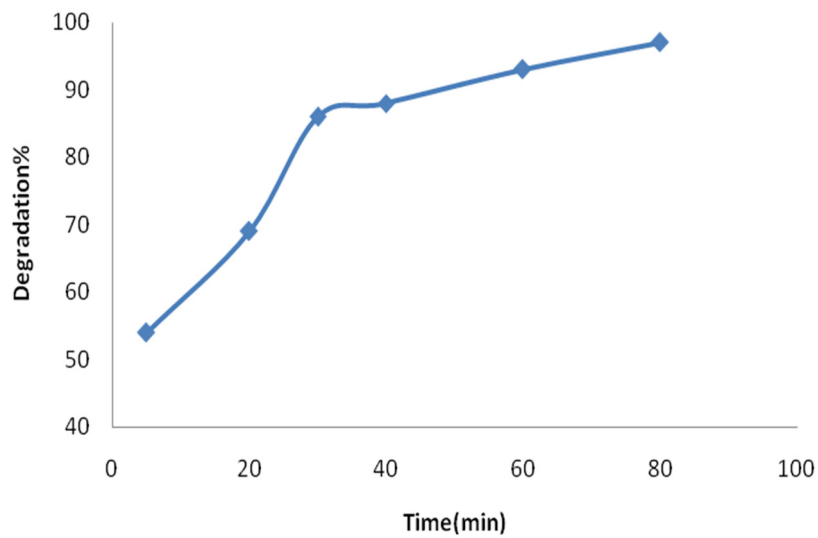


Fig. 9. Degradation percent of MB by MXene versus time under ultraviolet light irradiation

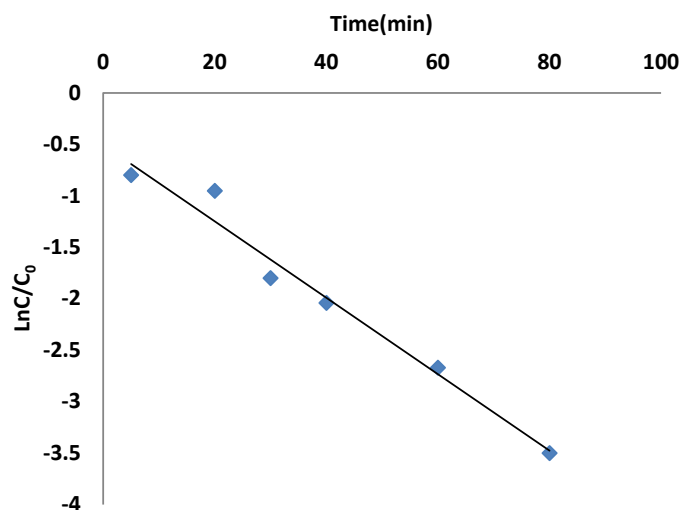


Fig. 10. $\ln C/C_0$ versus time in presence of 20 mg of MXene in samples of 20 mg/L of MB under ultraviolet light irradiation.

$$\ln(C_t/C_0) = k_t \quad (2)$$

C_0 is the initial concentration of MB before irradiating, C_t is the concentration of MB after irradiating, K shows the apparent rate constant and t stands for the irradiation time.

Dye degradation was performed at optimum conditions in the presence of MXene. As can be seen that the corresponding plots of $\ln(C_t/C_0)$ versus irradiation time was displayed in Fig. 10, and the corresponding apparent rate constant was calculated according to the Eq. (2). Plot of $\ln(C_t/C_0)$ against time exhibit linear trends, which indicates that the MB photodegradation process is acceptably described by the pseudo-first-order model. Linear regression was employed to measure the reaction rate constant from the slope of the fitted curve by means of and the calculated value is 0.0372 1/min.

Considering the importance of the reusability of a catalyst due to the significant cost of reduction in dye treatment, the potential of regeneration and reuse of nanoparticles was studied by washing the dye absorbent with 10.0 mL of DDW, and it was found that the adsorbent could be reused after this procedure. Hence, the separated catalyst from a reaction solution should be repeatedly washed with deionized water and dried. A second run of the photodegradation of MB can then be carried out using the recovered catalyst. The cyclic performance of the photocatalyst shows 97.1%, 91.4% and 89.1% of MB dye degradation, in the 1st, 2nd and 3rd cycles after 80 min of irradiation, respectively. (Fig.11).

The reduction in the degradation efficiency of the photocatalyst is probably due to the loss of the photocatalysts mass during each cycle. The results showed that the activity of $Ti_3C_2(OH)_2$ MXene only decreased after three runs. Thus, it can be recommended as a promising photocatalyst for wastewater treatment.

The photocatalytic degradation of organic dyes by semiconductor catalyst under light irradiation is typically based on the excitation of the semiconductors by light irradiation to form photogenerated electron/hole pairs (Abdel-Ghani et al., 2019). Developed in the 20th century, photocatalysis has been rendered an environment-friendly technology. Upon the absorption of light by some special semiconductors, namely "photocatalysts", the excitation of electrons (e^-) originally in the valence band (VB) to the conduction band (CB) occurs which results in holes (h^+) in the initial position. Free electrons with strong reducibility can reduce the valence state of some elements in compounds (Li et al., 2021).

The reaction of rich electrons in the conduction band with adsorbed oxygen molecules,

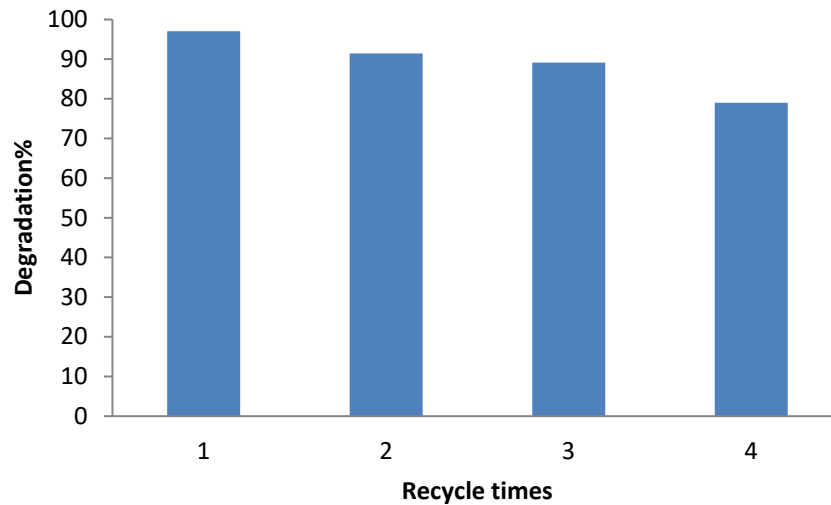


Fig. 11. The reusability tests of $\text{Ti}_3\text{C}_2(\text{OH})_2$ MXene photocatalyst for four cycles of MB degradation

Table 1. Comparison of the maximum degradation of MB onto various photocatalysts

Ref.	Degradation efficiency(%)	Degradation time(min)	Dye concentration (mg/L)	Dark reaction	Light source irradiation	Photocatalyst
(Belachew et al., 2020)	99	120	10	60	UV light	rGO-Ag/ZnO
(Yang et al., 2016)	96	150	10	30	Vis light	TiO ₂ /graphene porous
(Ke et al., 2017)	90	120	10	-	-	CQD/TiO ₂
(Udayabhaskar et al., 2019)	76	90	10	-	Vis light	Graphene/Cuo nanostructure
(Mohamed and Alsanea 2020)	99.9	60	20	30	UV	TiO ₂ /CDs/rGO
This work	97	80	20	20	UV	Ti ₃ C ₂ (OH) ₂ nanosheets

produces superoxide radicals (O_2^-). The holes generated migrate to the surface and thus react with water molecules to produce hydroxide radical (OH^\cdot) (Din et al., 2021). Then super oxide hydroxyl radicals were generated through protonation of radical anions ($\text{O}_2^{\cdot-}$). Finally, these radicals are responsible for degradation of organic pollutants.

The effect of different adsorbents on the photodegradation of MB have been studied to validate the photocatalytic performance of MXene. Photodegradation percentage of as-produced MXene was compared with other recent reports of photocatalysts for MB (Table 1). The results indicate that this adsorbent has great potential application in dye photo degradation from aqueous solution.

CONCLUSIONS

This study focuses on the direct application of MXene for the absorptive-photocatalytic degradation of MB. A simple and effective strategy was successfully used to design single layered

Ti₃C₂(OH)₂ nanosheets as an environmental-friendly photocatalyst. After 80 min of ultraviolet light exposure, outstanding degradation efficiency of Methylene Blue dye in the solution was established, which indicates that the MB photodegradation process is acceptably described by the pseudo-first-order model. Study the impact of contact time on degradation indicates that the MB photodegradation process is acceptably described by the pseudo-first-order model and rate constant (k) was calculated to be 0.0372 1/min. Methylene Blue dye in the solution was degraded by 97% after exposure to UV light. Also the produced MXene photocatalyst showed a good dispersibility in water and reusability up to 3 times without considerable deactivation. According to this study, the proposed photocatalysts can be reliably used for large-scale degradation of other toxic dyes from water and provide a notable example for future applications.

GRANT SUPPORT DETAILS

The present research did not receive any financial support.

CONFLICT OF INTEREST

The authors declare that there is not any conflict of interests regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/ or falsification, double publication and/or submission, and redundancy has been completely observed by the authors.

LIFE SCIENCE REPORTING

No life science threat was practiced in this research.

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