

**2D Ti₃C₂ MXene AND TiO₂/Ti₃C₂ HETEROSTRUCTURE: SYNTHESIS,
CHARACTERIZATION, AND APPLICATION IN WASTEWATER TREATMENT
VIA METHYLENE BLUE ADSORPTION**

A DISSERTATION

**SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE
AWARD OF THE DEGREE**

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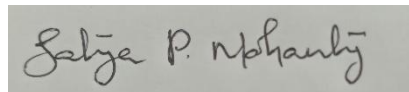
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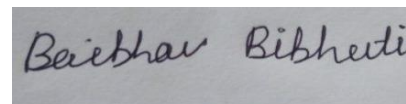
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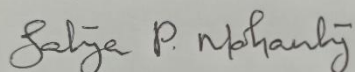
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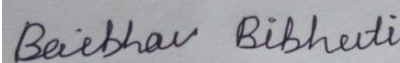
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ABSTRACT

The escalating discharge of toxic, non-biodegradable dyes from industrial activities necessitates advanced remediation strategies. This study synthesizes two-dimensional $\text{Ti}_3\text{C}_2\text{T}_x$ MXene via hydrothermal etching of Ti_3AlC_2 MAX phase using NaBF_4/HCl , followed by delamination, and constructs a $\text{TiO}_2/\text{Ti}_3\text{C}_2$ heterostructure through $\text{NaOH}/\text{H}_2\text{O}_2$ treatment to evaluate their methylene blue (MB) adsorption efficacy. Structural characterization reveals successful Al-layer removal in MXene, evidenced by XRD peak shifts from 9.5° to 6.39° and FESEM/TEM-confirmed multilayered nanosheets. The MXene exhibits a high specific surface area, mesoporosity, and negative zeta potential of -31.8 mV, enabling 99.42% MB removal within 90 minutes via electrostatic interactions. In contrast, the $\text{TiO}_2/\text{Ti}_3\text{C}_2$ heterostructure, despite a larger surface area and 3D safflower-like TiO_2 morphology, shows reduced adsorption of 66.4%, due to diminished surface charges (-16.9 mV). XPS confirms terminal -O/-OH/-Cl groups in MXene, while BET analysis underscores pore structure advantages for rapid dye uptake. The heterostructure's inferior performance highlights MXene's superior standalone efficacy. This work underscores hydrothermally synthesized $\text{Ti}_3\text{C}_2\text{T}_x$ MXene as a potent, scalable adsorbent for water purification, with future potential in hybrid systems and compares its structural properties and dye adsorbing capacities with $\text{TiO}_2/\text{Ti}_3\text{C}_2$ heterostructure and explains why the hydrothermally synthesized pristine $\text{Ti}_3\text{C}_2\text{T}_x$ MXene is better towards synthetic dye removal than the sophisticated nanohybrids and heterostructures.

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LIST OF SYMBOLS AND ABBREVIATIONS

h-MXene	Hydrothermally synthesized MXene
2-D	2-dimensional
DI water	Deionised water
UV-Vis	Ultraviolet-Visible
MAX	$M_{n+1}X_nT_x$
MB	Methylene Blue
XPS	X-ray Photoelectron Spectroscopy
BET	Brunauer-Emmett-Teller
XRD	X-ray Diffraction
FESEM	Field Emission Scanning Electron Microscope
TEM	Transmission Electron Microscope
SAED	Selected Area Electron Diffraction

CHAPTER 1

INTRODUCTION AND OBJECTIVES

1.1 INTRODUCTION

MXenes represent a revolutionary class of two-dimensional (2D) inorganic materials composed of transition metal carbides, nitrides, and carbonitrides[1]. Discovered in 2011 at Drexel University, these materials bridge the gap between ceramics and nanomaterials, offering a unique combination of metallic conductivity, mechanical robustness, and tunable surface chemistry[2]. Derived from MAX phases-ternary layered precursors with the formula $M_{n+1}X_nT_x$ (where M = transition metal, A = group 13/14 element, and X = carbon/nitrogen)- MXenes are synthesized by selectively etching the 'Al' layer, leaving atomically thin sheets terminated with functional groups (-OH, -O, -F)[2]. Their versatility has propelled research across energy storage, environmental remediation, biomedicine, and electronics, positioning them as transformative materials for sustainable technologies. The discovery of MXenes emerged from collaborative work by Yury Gogotsi and Michel Barsoum, who sought to exfoliate MAX phases. The first MXene, $Ti_3C_2T_x$, was produced by hydrofluoric acid (HF) etching of Ti_3AlC_2 , yielding a layered "accordion-like" structure[3]. This breakthrough unlocked a new paradigm in materials science, with over 30 compositions (e.g., V_2CT_x , $Mo_2TiC_2T_x$) now synthesized.

Recent advancements include scalable synthesis techniques, such as hydrothermal and solvothermal methods, which refine particle size and surface chemistry[4]. For instance, hydrothermal treatment at 150°C produces nitrogen-doped $\text{Ti}_3\text{C}_2\text{T}_x$ quantum dots with tunable optical properties. Despite progress, challenges persist in achieving uniform defect-free layers and large-scale production[5]. MXenes exhibit a hexagonal lattice structure with the general formula $\text{M}_{n+1}\text{X}_n\text{T}_x$ where surface terminations (T_x) dictate reactivity whose key properties include electrical conductivity which is upto 20,000 S/cm, rivaling graphene, due to delocalized d-electrons in transition metals, mechanical Strength: Young's modulus exceeding 300 GPa, enabling integration into flexible electronics, hydrophilicity: Water dispersibility facilitated by -OH/-O groups, critical for biomedical and environmental applications, thermal Stability: Retain structure up to 1,000°C in inert environments, with thermal conductivity reaching 55.8 W/(m·K) for $\text{Ti}_3\text{C}_2\text{T}_x$ [6].

The surface chemistry of MXenes is highly tunable. Functionalization with polymers (e.g., polyethyleneimine) or metals (e.g., Au nanoparticles) enhances properties like catalytic activity or ion intercalation. For example, $\text{Ti}_3\text{C}_2\text{T}_x$ functionalized with polyvinyl alcohol (PVA) forms flexible membranes for water purification, while nitrogen doping improves lithium-ion storage capacity in batteries[7].

MXenes excel in energy storage due to their pseudocapacitive behavior and high surface area. As supercapacitors, $\text{Ti}_3\text{C}_2\text{T}_x$ films achieve volumetric capacitances of $1,500 \text{ F/cm}^3$, outperforming carbon-based materials[8]. Their layered structure enables rapid ion diffusion, with 92% capacitance retention after 10,000 cycles. As batteries, Nb_2CT_x anodes deliver 420 m Ah/g in lithium-ion batteries, while MXene-coated separators mitigate polysulfide shuttling in lithium-sulphur systems[9]. Doping with transition metals (e.g., Mo) enhances theoretical capacities to 745 m Ah/g . For the generation of solar Energy, MXene/ TiO_2 hybrids drive hydrogen evolution at rates of 12 mmol/g/h under UV-vis light, leveraging their high photothermal conversion efficiency[9]. In the field of environmental remediation, MXenes' hydrophilicity and surface charge make them ideal for water treatment. For heavy metal removal, $\text{Ti}_3\text{C}_2\text{T}_x$ adsorbs Pb^{2+} at $1,200 \text{ mg/g}$, while composites with g- C_3N_4 degrade 98% of methylene blue under visible light[10]. For desalination purpose lamellar $\text{Ti}_3\text{C}_2\text{T}_x$ membranes reject 97% of divalent ions (e.g., Mg^{2+}) via size exclusion and electrostatic repulsion. Janus membranes combining $\text{Ti}_3\text{C}_2\text{T}_x$ with WS_2 exhibit threefold higher water permeance than conventional materials. As sensors, $\text{Ti}_3\text{C}_2\text{T}_x$ detects NH_3 at 1 ppm through resistivity changes, with potential for air quality monitoring. MXenes' biocompatibility and optical properties drive biomedical advances such as drug delivery, pH-responsive $\text{Ti}_3\text{C}_2\text{T}_x$ -PVA hydrogels enable controlled release of doxorubicin, reducing off-target toxicity[11].

For photothermal Therapy, $\text{Ti}_3\text{C}_2\text{T}_x$ converts near-infrared light to heat, achieving 90% tumor ablation in vivo. Gold-MXene hybrids enhance contrast in computed tomography and photoacoustic imaging[12].

MXene composites inhibit bacterial growth (e.g., *E. coli*) through physical disruption and reactive oxygen species generation which make them a useful candidate for antibacterial coatings[13]. Due to their excellent conductivity and flexibility, MXenes, it enable next-generation electronics and Optoelectronic devices. As EMI Shielding $\text{Ti}_3\text{C}_2\text{T}_x$ films ($2.5\ \mu\text{m}$) attenuate 92 dB of electromagnetic interference, surpassing metals[14]. As flexible Sensors: MXene/PDMS composites exhibit a gauge factor of 500, ideal for wearable strain sensors. Blade-coated $\text{Ti}_3\text{C}_2\text{T}_x$ films act as transparent electrodes, achieve 83% transmittance with 19,325 S/cm conductivity, rivaling indium tin oxide (ITO) in solar cells and touchscreens[15].

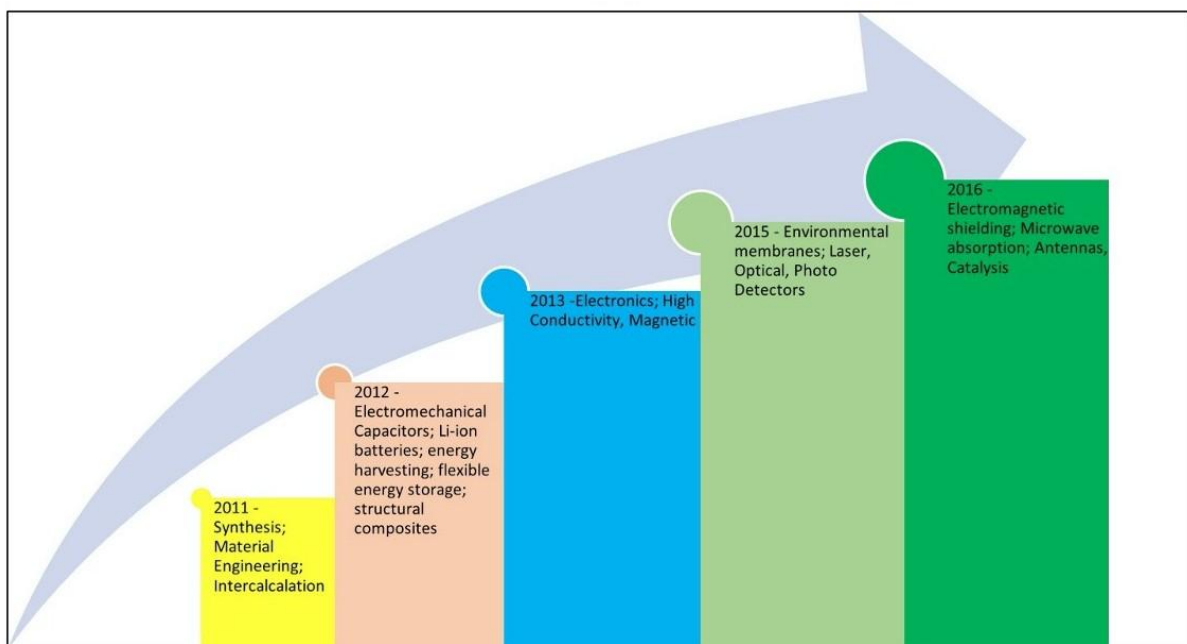


Fig. 1 $\text{Ti}_3\text{C}_2\text{T}_x$ MXene application history

MXenes are synthesized through diverse methods that balance safety, scalability, and material performance. These methods broadly fall into top-down etching, bottom-up synthesis, and hybrid approaches, each offering unique advantages and challenges[15]. The most common route involves selectively removing the "Al" layer (e.g., Al, Ga) from MAX phases ($M_{n+1}AX_n$) using Hydrofluoric Acid (HF) Etching: Direct etching of MAX phases (e.g., Ti_3AlC_2) with concentrated HF yields MXenes like $Ti_3C_2T_x$. [16] While effective, HF's toxicity and corrosivity necessitate stringent safety protocols, including fume hoods and protective gear. Safer alternatives use HCl/LiF mixtures to generate HF in situ, reducing hazards while enabling lithium intercalation for delamination[17]. Another method is molten Salt Etching, by employing salts like $ZnCl_2$ or $SnCl_2$ at high temperatures (550–873°C) avoids HF entirely. This method introduces tunable surface terminations (e.g., Cl, S) and produces stable MXenes, though it requires energy-intensive conditions. Electrochemical Etching is another similar method where by utilizing electrolytes like HBF_4 , this HF-free method selectively dissolves the 'Al' layer via anodic oxidation[18]. It scales efficiently and yields MXenes with larger lateral dimensions, ideal for supercapacitors.

There are also bottom-up synthesis methods of bypassing MAX phases, constructing MXenes atom-by-atom. One such method is Chemical Vapor Deposition (CVD), which involves reacting metal halides (e.g., $TiCl_4$) with carbon/nitrogen sources (e.g., CH_4 , N_2) on substrates produces high-purity MXenes. CVD enables vertical alignment (e.g., Ti_2CCl_2 "carpets") and novel morphologies like vesicles, enhancing applications in catalysis and energy storage.

Hydrothermal/Solvothermal synthesis involves aqueous or solvent-based reactions under high pressure/temperature (e.g., NaOH etching of $\text{Mo}_2\text{Ga}_2\text{C}$) yield MXene quantum dots (QDs) for bioimaging, though reaction times are prolonged[19]. Recent research follows some hybrid and advanced methods like mechanochemical Pretreatment: ball milling MAX phases before etching increases interlayer spacing, improving etching efficiency and electrochemical performance[20]. Template-assisted assembly: 3D-printed templates guide MXene aerogel formation via cation-induced gelation, creating architectures for high-capacity energy storage[21].

With surge in rapid industrialization across the globe, amount of air and water pollution also has been increased exponentially. The textile, leather, and paper industries are major contributors to global water pollution, releasing billions of liters of dye-contaminated wastewater annually[22]. Synthetic dyes, such as azo, anthraquinone, and triarylmethane compounds, are chemically stable, non-biodegradable, and often toxic. Approximately 10–15% of dyes used in industrial processes are discharged untreated into water bodies, contaminating aquatic ecosystems and threatening biodiversity[23]. These dyes block sunlight penetration, impairing photosynthesis in algae and aquatic plants, which disrupts oxygen levels and destabilizes aquatic food chains. Additionally, dye effluents elevate biochemical oxygen demand (BOD) and chemical oxygen demand (COD), depleting dissolved oxygen and causing hypoxia, which suffocates marine life. Heavy metals like lead, chromium, and cadmium, frequently present in industrial

effluents, exacerbate toxicity, leading to bioaccumulation in fish and eventual entry into the human food chain[24]. Chronic exposure to these pollutants is linked to cancers, organ failure, and neurological disorders. Soil irrigated with contaminated water suffers reduced fertility, altered microbial ecosystems, and inhibited crop growth, undermining agricultural sustainability[25].

Conventional remediation methods, such as activated carbon adsorption, chemical precipitation, and biological treatments, face limitations. Activated carbon is costly and inefficient for low-concentration dyes, while chemical methods generate toxic sludge[26]. Biological processes struggle with dye recalcitrance, and advanced oxidation techniques are energy-intensive. MXenes—emerging two-dimensional materials composed of transition metal carbides, nitrides, or carbonitrides—offer a groundbreaking solution. Synthesized by selectively etching the "Al" layer from MAX phases (e.g., Ti_3AlC_2), MXenes like $\text{Ti}_3\text{C}_2\text{T}_x$ exhibit a layered structure, high surface area ($\sim 1,500 \text{ m}^2/\text{g}$), and tunable surface terminations (-OH, -O, -F), making them exceptional adsorbents[27].

MXenes excel in dye removal due to their negatively charged surfaces, which electrostatically attract cationic dyes (e.g., methylene blue, malachite green) and form hydrogen bonds with organic pollutant[28]. For instance, $\text{Ti}_3\text{C}_2\text{T}_x$ MXene achieved 99.1% removal of malachite green within 150 minutes, while MXene- Co_3O_4 composites demonstrated a 136.24 mg/g adsorption capacity for methylene blue, surpassing conventional adsorbents[29]. Functionalization with polymers

(e.g., chitosan) or nanoparticles (e.g., ZnO) enhances selectivity and efficiency. MXene/ZnO hybrids removed 97% of Pb^{2+} and As^{3-} via synergistic effects, showcasing versatility in addressing mixed contaminants[30]. The dye removal efficiencies of different adsorbent [31]materials have been noted in Table 1. MXenes also address challenges like reusability and scalability. Regeneration via thermal treatment or solvent washing retains more than 90% efficiency after multiple cycles[32]. Green synthesis methods, such as molten salt etching (e.g., ZnCl_2/KCl), avoid toxic hydrofluoric acid (HF), reducing environmental hazards. Their mechanical flexibility enables integration into membranes for continuous filtration systems[33]. For example, $\text{Fe}_3\text{O}_4/\text{Ti}_3\text{C}_2$ composites combine magnetic separation with high adsorption, simplifying recovery and reuse[34].

In this work, we have synthesized 2D MXene $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheets using a hydrothermal route from precursor MAX phase MAX-phase Ti_3AlC_2 . In the next step a unique safflower shaped $\text{TiO}_2/\text{Ti}_3\text{C}_2$ heterostructure with three-dimensional (3D) porous frameworks was synthesized from $\text{Ti}_3\text{C}_2\text{T}_x$ MXene[35]. The synthesized $\text{Ti}_3\text{C}_2\text{T}_x$ MXene and $\text{TiO}_2/\text{Ti}_3\text{C}_2$ were subjected to different material characterizations, and their structural properties were investigated[36]. Further we have performed dye adsorption[37] test on both the synthesized materials and and based on the results, a comparative analysis on their adsorbance property is done[38].

Table 1. Different materials and their dye removal efficiencies

S.no.	Material	Morphology	Dye	Dye removal efficiency
1	SnO ₂ / MXene	Bulk structure to form sheets where both SnO ₂ & MXene are agglomerated.	MB	70% for 120 mins under direct sunlight.
2	TiO ₂ / MXene	Layered of Ti ₃ C ₂ MXene and an array of TiO ₂ particles on the external MXene surface.	MO	99% for 40 mins under light irradiation.
3	TiO ₂ /MXene	Spherical TiO ₂ particle uniformly supported on MXene nanosheet.	MB	96.44% in UV light for 60 mins.
4	ZnO/ MXene	ZnO particle dispersed over the loose accordion-shape layered structure with smooth surface. At higher temperature ZnO micro-rods & twinned ZnO structure.	RhB	97.5% with 18 mins under UV light.
5	ZnO/ MXene	Flower like ZnO loaded on the surface of MXene in the form of rods.	MB	94.8% with 180 mins in visible light.
6	ZnO/ RGO	RGO sheet are exfoliated and intact with ZnO providing a composite material	CR	98% for 90 mins with (PH-10) alkaline.
7	TiO ₂ /RGO	TiO ₂ NPs	RhB	94.55% for 120 mins in light.
8	ZnO/SnO ₂ /RGO	SnO ₂ /ZnO-core shell nanostructure RGO- nanosheet	CR	Orange-II = 99.8% reactive red = 97.02 for 120 mins under visible light.
9	SnO ₂ /gC ₃ N ₄	Condensed layer of porous gC ₃ N ₄ covering the surface of SnO ₂	RhB	99.42% for 150 mins under led-30 W.
10	Cu-ZnO/gC ₃ N ₄	Wurzite structure bundles with hexagonal nanorods	RhB MB	98% & 99% for 40 mins under UV light.

11	TiO ₂ /gC ₃ N ₄	TiO ₂ nanoparticle on the surface of gcn nanosheet	Sodium diclofenac	99.99% for 120 mins with scavenger.
12	Ta (3%) -WO ₃	WO ₃ -irregularly shaped rough nano spheroid. Ta/WO ₃ -sharp and porous nano cubes of diff. nm. range.	MB	91% IN 120mins under 400 W power metal halide lamp.
13	Ta (3%)-TiO ₂	TiO ₂ NPs	RhB MB	91% & 88% for 120mins under UV light.
14	WO ₃ /Ti ₃ C ₂	2D-2D nanosheets	MB	92%, for 105mins

CHAPTER 2

EXPERIMENTAL

2.1 Material and Reagents

37 wt% Sodium tetraborofluorate (NaBF_4) provided by Vizak Chemicals. 30% HCl was bought from Green Agri Solution. Ti_3AlC_2 (MAX phase) was purchased from Aritech Chemazone pvt. Ltd. 1M NaOH solution and 30% Hydrogen Peroxide (H_2O_2) was bought from Annexe Chem pvt. Ltd. Methylene Blue (MB) provided by Jalan Dyestuff Co.

2.2 SYNTHESIS METHODS

2.2.1 Hydrothermal synthesis of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene

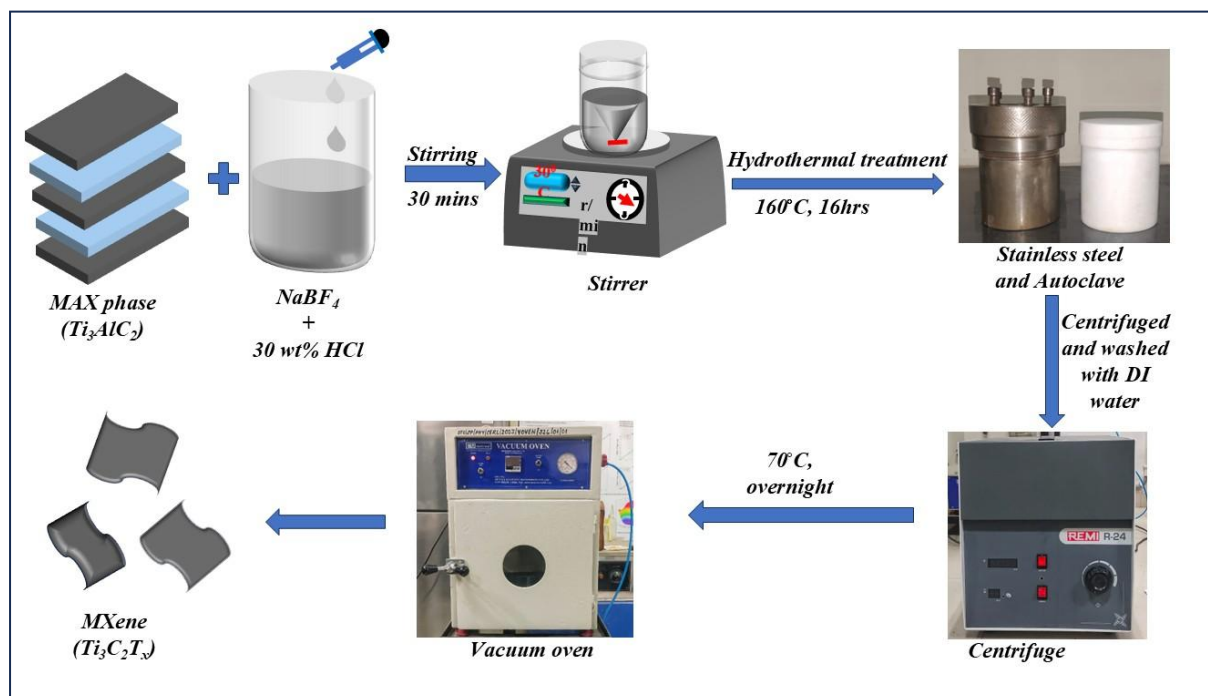


Fig. 2 Schematic representation of hydrothermal synthesis of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene

The hydrothermal synthesis of h-Ti₃C₂ MXene involves a series of precise chemical steps. Sodium tetrafluoroborate (NaBF₄) is dissolved in 37 wt% hydrochloric acid (HCl). To this solution, titanium aluminium carbide (Ti₃AlC₂) is added and the mixture is stirred until uniform. This suspension is then transferred to a 100 mL autoclave, ensuring that it is filled to no more than 30% of its capacity, hydrothermally treated at 180 °C for a duration ranging from 8 to 32 hours[39].

2.2.2 Synthesis of TiO₂/Ti₃C₂ heterostructure

The one-step synthesis method for TiO₂/Ti₃C₂ heterostructures can be effectively performed using a hydrothermal approach. In this process, Ti₃C₂T_x MXene is used as both the substrate and titanium source. First, 200 mg of Ti₃C₂ powder is dispersed in deionized water to form a suspension. This suspension is then treated with 60 ml of 1 M NaOH and 4 ml of 30% H₂O₂, which is added to the mixture. The resulting solution is transferred to a Teflon-lined autoclave and subjected to hydrothermal treatment at 160 °C for 12 hours. This treatment facilitates the growth of TiO₂ directly on the Ti₃C₂ surface, forming a heterojunction that enhances photocatalytic properties. After cooling, the product can be washed and dried to obtain the TiO₂/Ti₃C₂ heterostructure[40], [41].

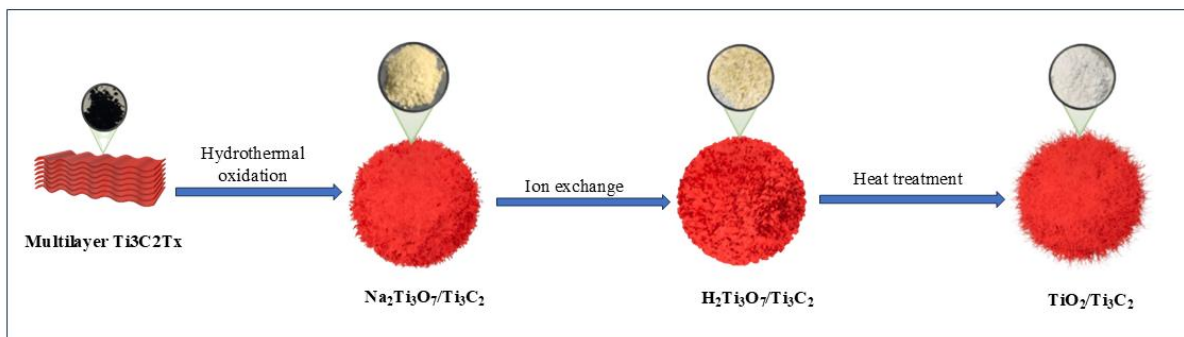


Fig. 3 Schematic representation of stepwise synthesis of TiO₂/Ti₃C₂

2.3 Characterization Techniques

The crystal structure of the synthesized $\text{Ti}_3\text{C}_2\text{T}_x$ MXene and $\text{TiO}_2/\text{Ti}_3\text{C}_2$ heterostructure was studied using X-ray Diffraction (XRD, Rigaku MNCF 030) with $\text{Cu K}\alpha 1$ and $\text{Cu K}\alpha 2$ radiation. Surface morphology was investigated using a field emission scanning electron microscope (FESEM, FEI NOVA NANOSEM-450) with energy dispersive x-ray spectrometer (EDX) mounted. TEM, HRTEM and SAED pattern study reveals the detailed structure, fringe pattern, grain boundary and atomic arrangement using a transmission electron microscope (TEM, JOEL/JEM-F200). Brunauer-Emmett-Teller (BET, BELSORP max II) study was carried out to find out the specific surface area by N_2 adsorption-desorption curve. The surface elemental analysis and binding energies were determined by X-ray photoelectron spectrometer (XPS, Kratos Analytical MNCF 026) with $\text{Al K}\alpha$ radiation. Zeta potential for both the samples was evaluated using Malvern Panalytical zetasizer.

2.4 Dye adsorption test

To test the adsorption capacity of $\text{Ti}_3\text{C}_2\text{T}_x$ and $\text{TiO}_2/\text{Ti}_3\text{C}_2$, dye adsorption test was conducted, for which MB was taken as the main probing dye. A stock solution was prepared whose concentration is taken to be $31 \mu\text{M}$ (1mg in 100 ml of deionized water). The adsorbent amount was taken to be 25 mg. In order to avoid any photocatalytic effect, the whole setup was kept inside dark environment. The rate of MB dye removal efficiency was evaluated using the following formula

$$R (\%) = [(A_o - A_t) / A_o] \times 100$$

CHAPTER 3

RESULTS AND DISCUSSIONS

3.2.1. XRD

Fig. 4 is the XRD pattern which reveals the crystallinity and phase structure of the synthesized $\text{Ti}_3\text{C}_2\text{T}_x$ MXene and $\text{TiO}_2/\text{Ti}_3\text{C}_2$ heterostructure. The precursor Ti_3AlC_2 (MAX phase) shows characteristic peaks at 9.5° , 19.1° and 39.1° [42] which corresponds to the (002), (004) and (104) planes (JCPDS 52-0875). When, it is subjected to hydrothermal etching, the Al layer gets removed. As a result the interplanar distance increases.

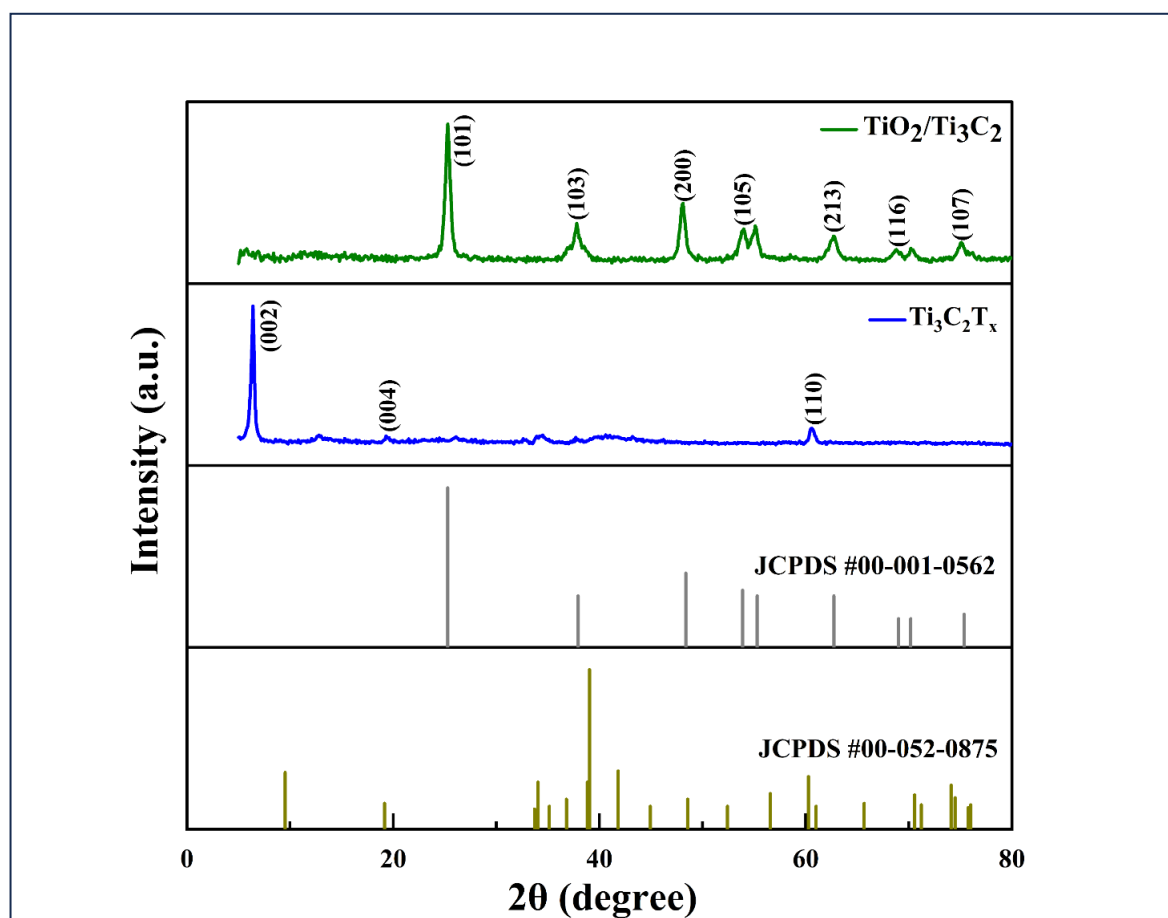


Fig. 4 XRD pattern of $\text{Ti}_3\text{C}_2\text{T}_x$ and $\text{TiO}_2/\text{Ti}_3\text{C}_2$

This can be observed from the XRD pattern that the most intense peak at 39.1° correspond to (104) plane of Ti_3AlC_2 has been completely vanished and the peaks at 19.1° and 60.2° which correspond to (004) and (110) planes have been diminished and the peak at 9.5° has been shifted to 6.39° [43]. This confirms the formation of MXene from the MAX phase. Further when the $\text{Ti}_3\text{C}_2\text{T}_x$ MXene was subjected to hydrothermal oxidation, followed by ion exchange and heat treatment, a drastic change in the XRD pattern was observed[44]. New peaks were observed at 25.3° , 38.1° , 48.4° , 53.9° , 62.7° , 69° , 75.4° which correspond to (101), (103), (200), (105), (213), (116), (107) diffraction planes of anatase TiO_2 (JCPDS 01-0562)[45]. The d-spacing was calculated to be 13.83\AA and 3.52\AA for $\text{Ti}_3\text{C}_2\text{T}_x$ MXene and $\text{TiO}_2/\text{Ti}_3\text{C}_2$ heterostructure respectively. Further using debye-scherrer's formula, crystallite size of both the samples were determined to be 18.15nm and 20.37 nm [46], [47].

3.2.2 FESEM with EDX mapping

The surface morphology of the synthesized $\text{Ti}_3\text{C}_2\text{T}_x$ MXene and $\text{TiO}_2/\text{Ti}_3\text{C}_2$ heterostructure were examined using a field-emission scanning electron microscope (FEI, NOVA NANOSEM-450). It can be clearly observed from Fig. 5(a, b) that the randomly arranged MAX phase have been successfully transformed into multilayered MXene nanosheets, which further confirms the etching of Al layers. Some small granules can be observed in the FESEM image, which are due to incomplete transformation of MAX phase to $\text{Ti}_3\text{C}_2\text{T}_x$ [48], [49]. This could be achieved by increasing the hydrothermal etching time but by doing so chances of agglomeration would be high. Fig. 5(c, d) are the FESEM images of $\text{TiO}_2/\text{Ti}_3\text{C}_2$ heterostructure, where

3D safflowers of TiO_2 has been emerged out of the 2D MXene which well resembles with previously reported literature[40], [41].

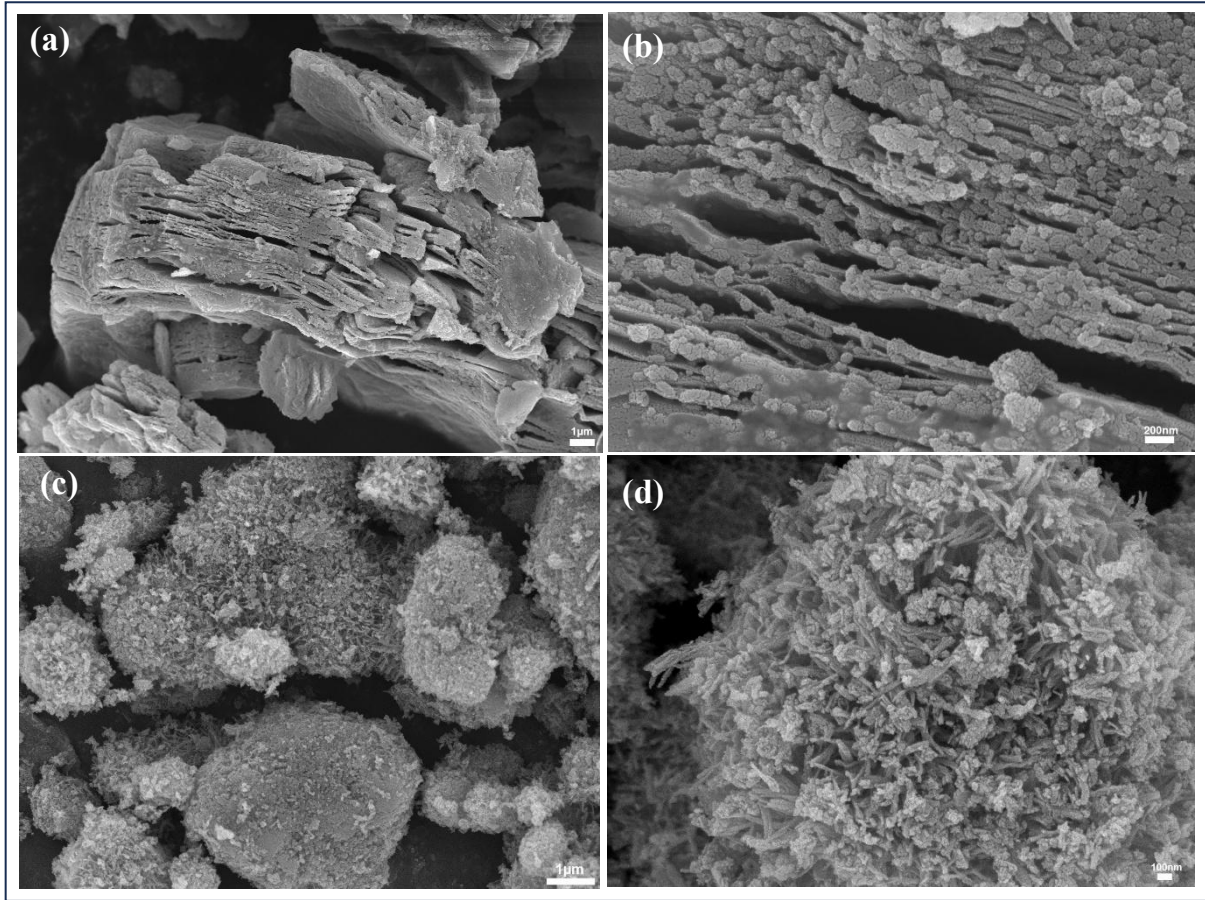


Fig. 5 FESEM images of (a,b) $\text{Ti}_3\text{C}_2\text{T}_x$ and (c,d) $\text{TiO}_2/\text{Ti}_3\text{C}_2$

Electron dispersive x-ray spectrometry (EDX) is used to determine the chemical environment and elemental distribution in the synthesized material which can be seen in fig. 6. Table 2 depicts the elements present in $\text{Ti}_3\text{C}_2\text{T}_x$ and their atomic %, where (C: O:Cl:Ti::14.5:12.1:5.7:65.8) are present and there is no trace of Al, which suggests the etching of Al layer from the Ti_3AlC_2 MAX phase. Likewise, in the case of $\text{TiO}_2/\text{Ti}_3\text{C}_2$ heterostructure, there is only (C: O:Ti ::17.8:44.9:37.4) are present. Surface termination groups like oxides and halides are absent, which makes it less anionic species than $\text{Ti}_3\text{C}_2\text{T}_x$.

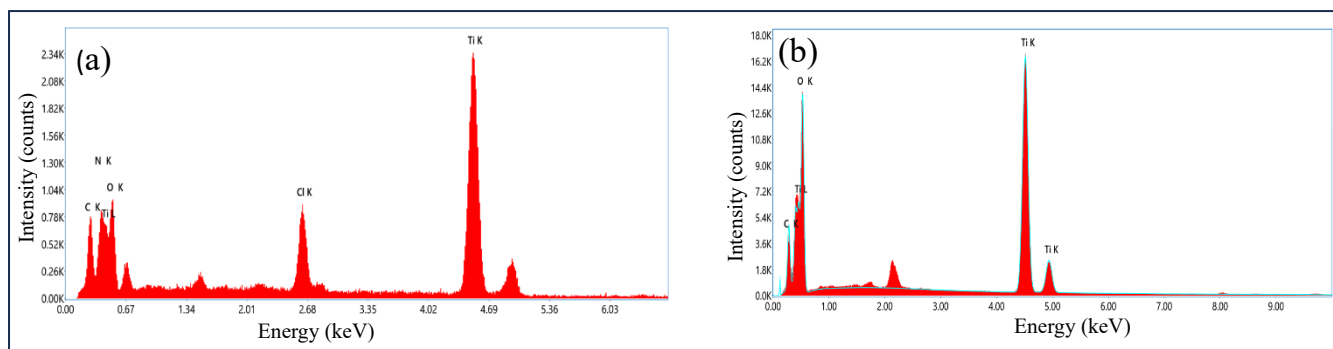


Fig. 6 EDX spectrum of (a) $\text{Ti}_3\text{C}_2\text{T}_x$ (b) $\text{TiO}_2/\text{Ti}_3\text{C}_2$

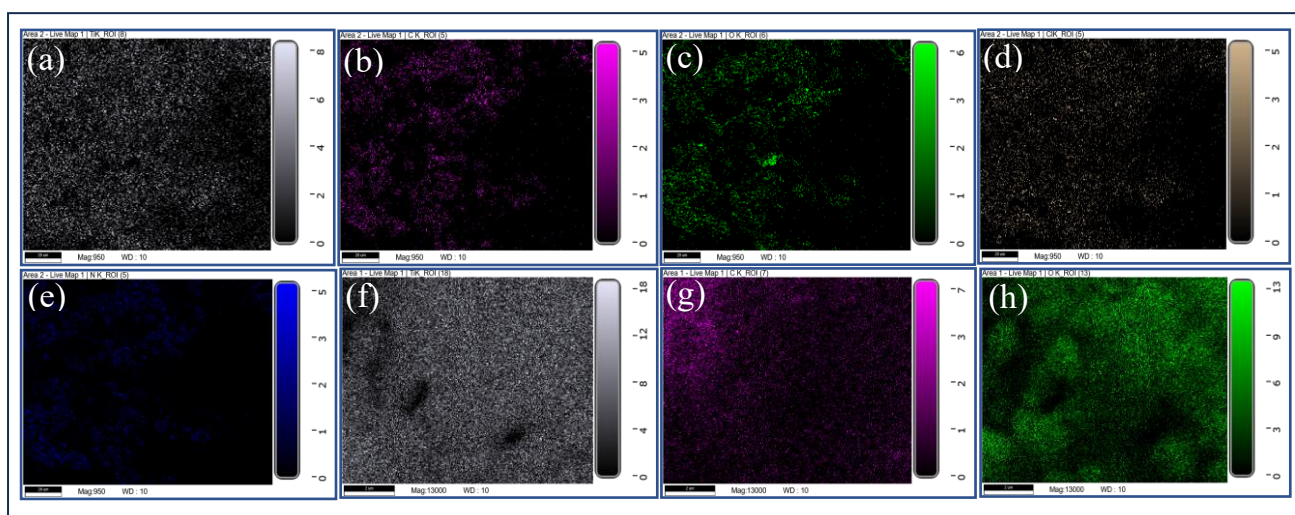


Fig. 7 EDX elemental mapping of (a-e) $\text{Ti}_3\text{C}_2\text{T}_x$ and (f-h) $\text{TiO}_2/\text{Ti}_3\text{C}_2$

Table 2. Elemental composition of (a) $\text{Ti}_3\text{C}_2\text{T}_x$ and (b) $\text{TiO}_2/\text{Ti}_3\text{C}_2$

(a)	Element	Weight %	Atomic %
	C K	14.1	32.1
	N K	3.1	6.0
	O K	11.7	20.1
	Cl K	5.7	4.4
	Ti K	65.4	37.4

(b)	Element	Weight %	Atomic %
	C K	17.8	29.2
	O K	44.9	55.4
	Ti K	37.4	15.4

3.2.3 TEM

The detailed microstructural view of the synthesized $\text{Ti}_3\text{C}_2\text{T}_x$ MXene and $\text{TiO}_2/\text{Ti}_3\text{C}_2$ heterostructure was viewed under transmission Electron Microscope (TEM) which shows the layered structure of $\text{Ti}_3\text{C}_2\text{T}_x$. High-resolution transmission electron microscopy (HRTEM) reveals lattice fringes of 0.31 nm, corresponding to the (100) planes of $\text{Ti}_3\text{C}_2\text{T}_x$, and confirms the presence of defect-free, ultrathin sheets. Selected-area electron diffraction (SAED) patterns exhibit hexagonal symmetry, validating the crystalline structure of MXene[50].

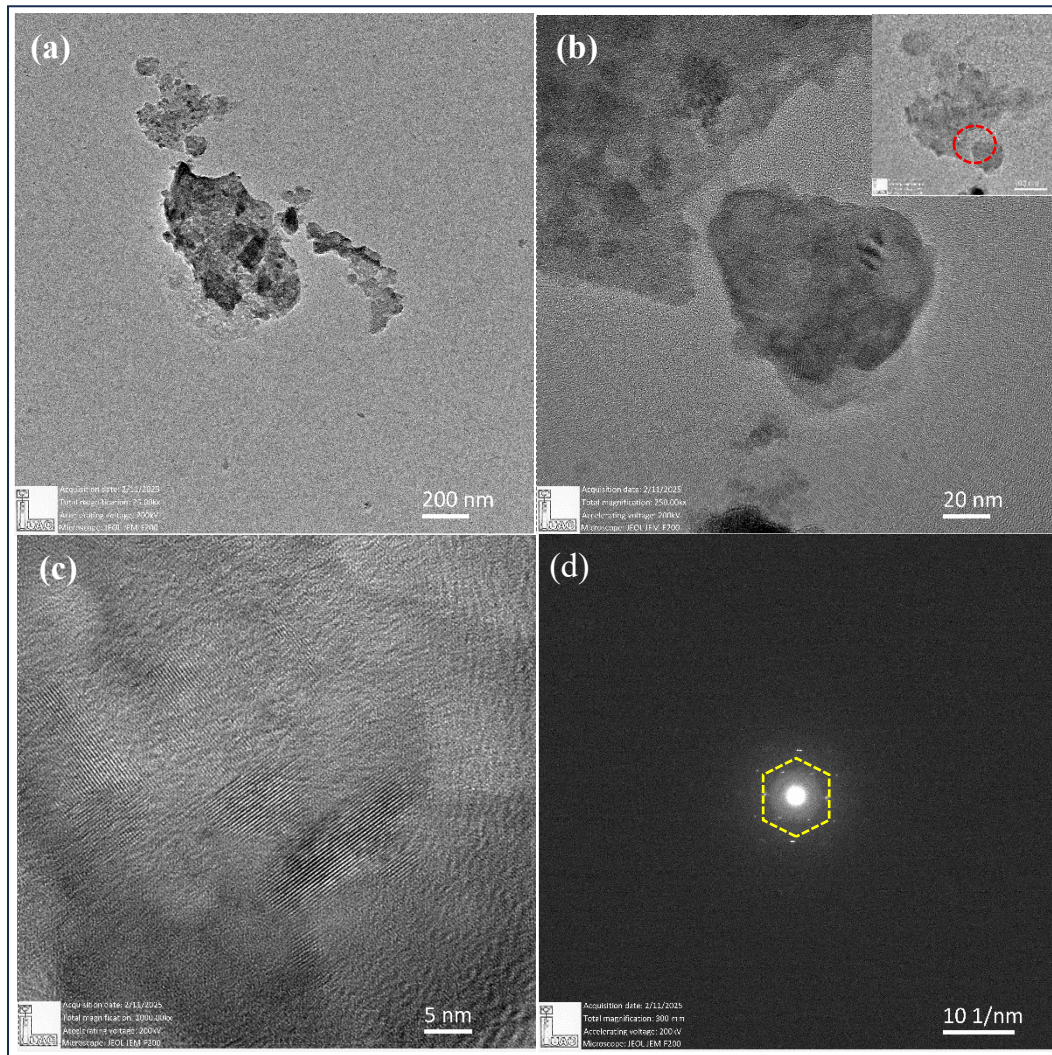


Fig. 8 (a, b) TEM, (c) HRTEM, (d) SAED pattern images of $\text{Ti}_3\text{C}_2\text{T}_x$

For $\text{TiO}_2/\text{Ti}_3\text{C}_2$ heterostructures, hydrothermal oxidation methods facilitate the growth of TiO_2 nanosheets on MXene surfaces. Cross-sectional TEM imaging shows interlayer spacing reductions from ~ 1.1 nm (pristine MXene) to ~ 0.35 nm post-oxidation, indicative of TiO_2 integration. HRTEM highlights epitaxial alignment between anatase TiO_2 (0.35 nm lattice spacing for (101) planes) and Ti_3C_2 , with SAED confirming composite diffraction rings from both phases. Elemental mapping via EDS underscores uniform Ti, C, and O distribution, while HAADF-STEM imaging visualizes surface functional groups and interfacial coherence[40], [41].

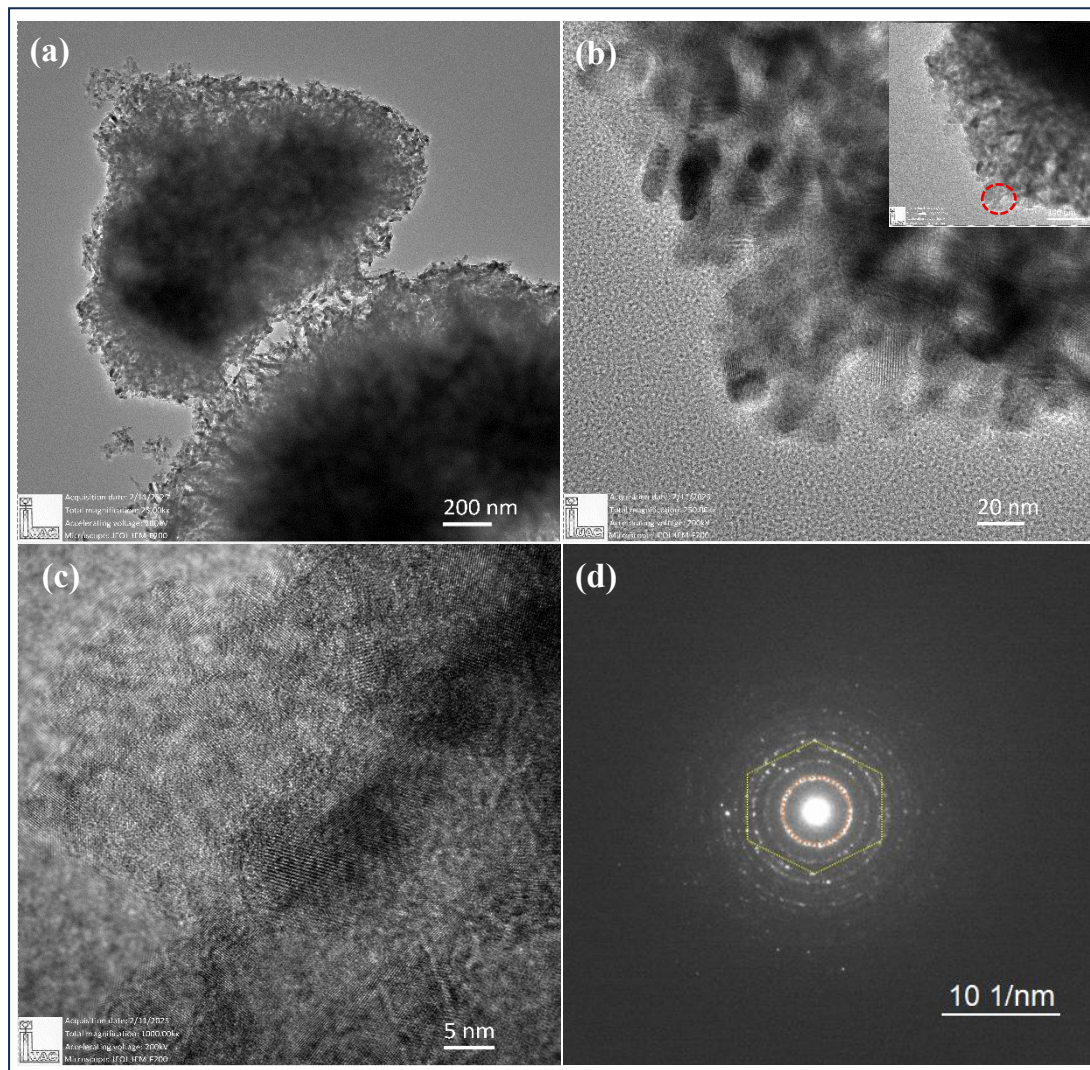


Fig. 9 (a, b) TEM, (c) HRTEM, (d) SAED pattern images of $\text{TiO}_2/\text{Ti}_3\text{C}_2$

3.2.4 XPS

X-ray photoelectron spectroscopy (XPS) is pivotal in elucidating the surface chemistry of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene. In $\text{Ti}_3\text{C}_2\text{T}_x$ MXene, survey spectra reveals the elemental composition which contains O 1s, Ti 2p, C 1s, F 1s, Cl 2p with at.% 37.7, 17.92, 32.47, 6.88, 5.03 and peak binding energy position at at 532 eV, 459.97 eV, 286.47 eV, 685.98 eV, 200.35 eV. High-resolution Ti 2p scan reveal distinct bonding states: Ti–C (456 eV), Ti–O (457 eV), Ti–F (458 eV), and TiO_2 (459 eV), indicating surface terminations (-O, -OH, -F, -Cl) and partial oxidation[50], [51].

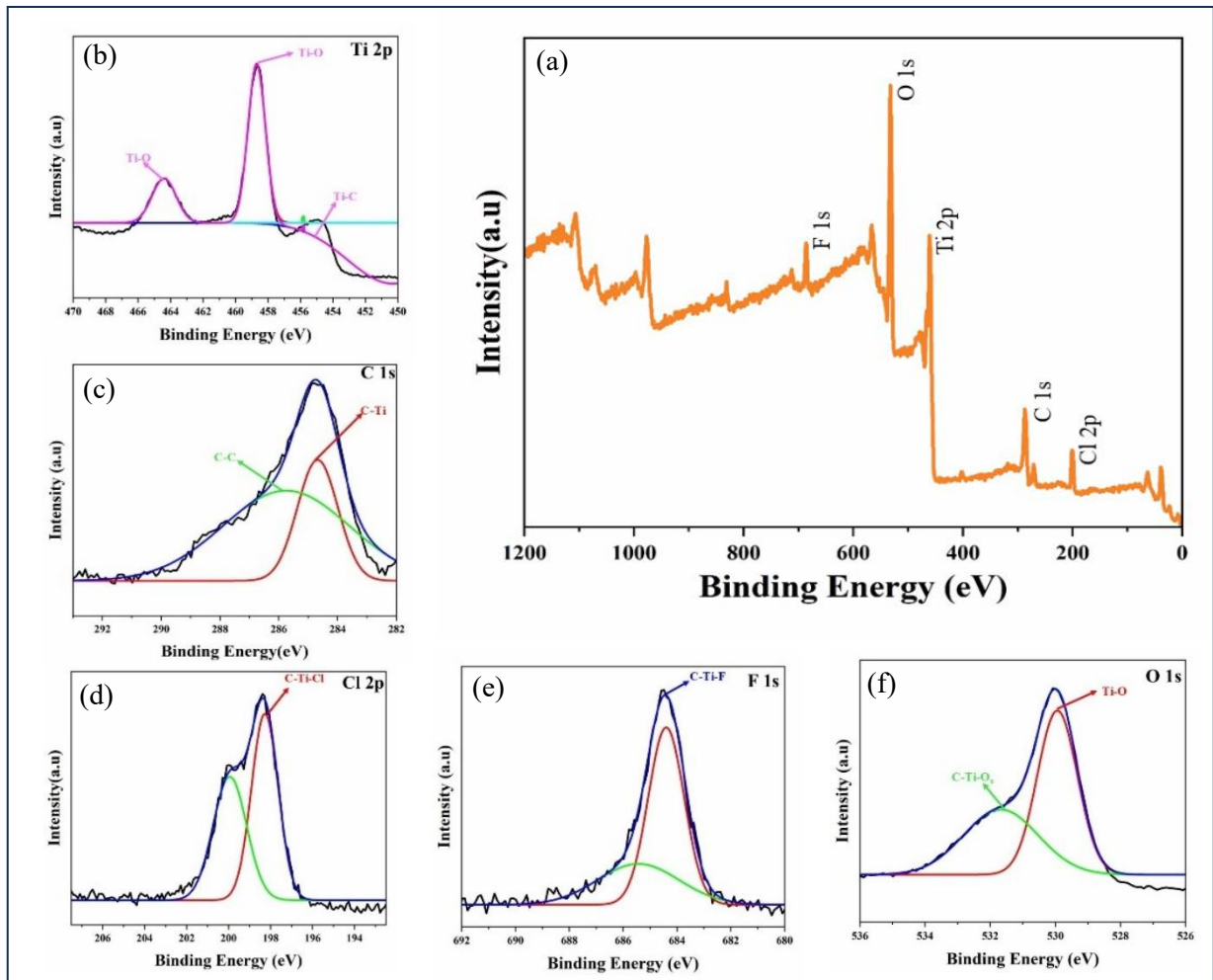


Fig. 10 (a) XPS survey spectra, (b-f) high resolution scan of Ti 2p, C 1s, O 1s, F 1s, Cl 2p of $\text{Ti}_3\text{C}_2\text{T}_x$

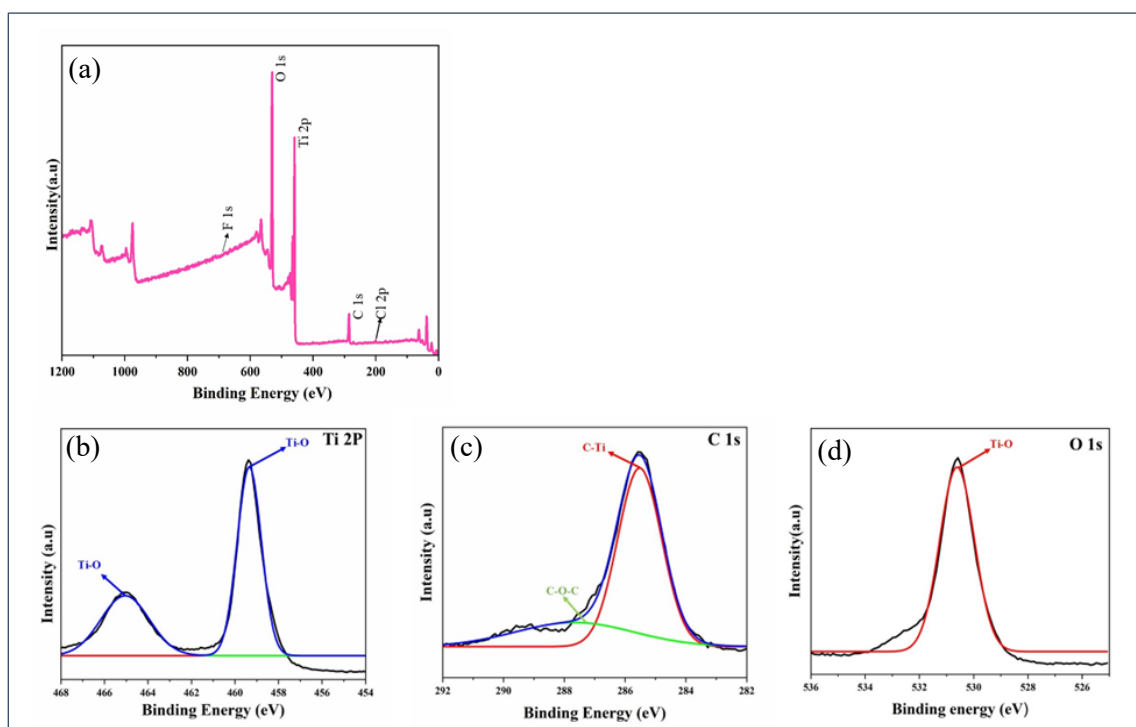


Fig. 11 (a) XPS survey spectra, (b-d) high resolution scan of Ti 2p, C 1s, O 1s of TiO₂/Ti₃C₂

The C 1s spectrum features Ti–C (282 eV) and adventitious carbon (284.8 eV), while O 1s peaks correspond to Ti–O (530 eV), Ti–OH (531.5 eV), and adsorbed H₂O (533 eV). F 1s spectra (686 eV) confirm Ti–F bonds. For TiO₂/Ti₃C₂ heterostructures, XPS highlights TiO₂ formation with Ti content (29.2 at.%) (Ti⁴⁺ at 459 eV) and reduced F content (0.15 at.%), and Cl content (0.99 at.%) suggesting almost absence of surface groups, enhancing its stability. The O 1s spectrum shifts toward lattice oxygen (530 eV), with residual Ti–OH and H₂O. Hierarchical structures, such as 1D TiO₂ nanowires on 2D Ti₃C₂, mitigate layer stacking and oxidation, confirmed by cross-sectional analysis. Surface chemistry reveals increasing O (44.6 at.%) and reducing C (24.5 at.%), while maintaining Ti–C integrity[40], [41].

3.2.5 BET

BET method is critical for evaluating the specific surface area (SSA) and porosity of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene and $\text{TiO}_2/\text{Ti}_3\text{C}_2$ heterostructure. For $\text{Ti}_3\text{C}_2\text{T}_x$, BET analysis reveals the SSA value is determined to be $153.63 \text{ m}^2/\text{g}$, with pore volumes of $0.27 \text{ cm}^3/\text{g}$ and average pore diameters of 16.8\AA [52]. Nitrogen adsorption-desorption isotherms for $\text{Ti}_3\text{C}_2\text{T}_x$ often conform to Type IV (IUPAC classification) isotherm with H3 hysteresis, indicative of slit-shaped mesopores formed by aggregated nanosheets. The Barrett-Joyner-Halenda (BJH) pore size distribution confirms narrow mesopore ranges, enhancing dye adsorption. For $\text{TiO}_2/\text{Ti}_3\text{C}_2$ heterostructures, BET surface area is determined to be $181.22 \text{ m}^2/\text{g}$, influenced by TiO_2 nanoparticle size and distribution[53]. The increased surface area of $\text{TiO}_2/\text{Ti}_3\text{C}_2$ may attributed to the 3D flower like structure of TiO_2 but the incorporation of TiO_2 introduces new mesopores with pore volumes of $0.31 \text{ cm}^3/\text{g}$ and average pore diameters of 18\AA which partially block MXene pores[54], [55]. So, further zeta potential test was conducted to determine the adsorbent action over MB.

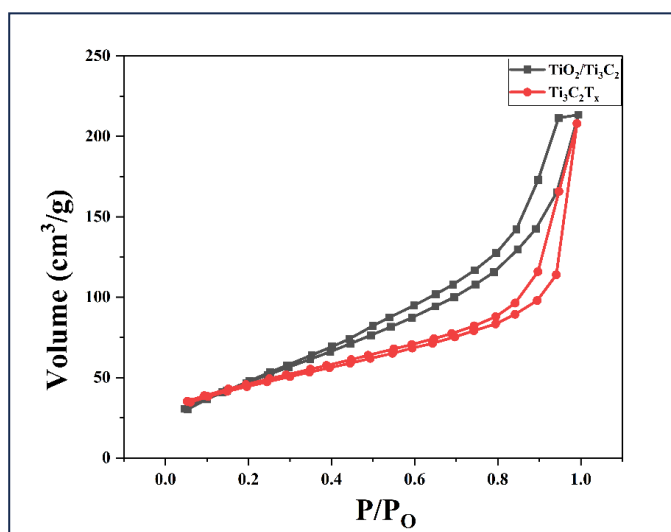


Fig. 12 N_2 adsorption-desorption isotherm for $\text{Ti}_3\text{C}_2\text{T}_x$ and $\text{TiO}_2/\text{Ti}_3\text{C}_2$

3.2.6 ZETA POTENTIAL

$\text{Ti}_3\text{C}_2\text{T}_x$ MXene being an anionic species exhibits a strongly negative surface charge before adsorption, with zeta potential value -31.8 mV in neutral pH conditions. This negativity is due to the presence of deprotonated surface groups ($-\text{O}^-$, $-\text{OH}^-$) and chlorine terminations ($-\text{Cl}^-$). Following methylene blue (MB) adsorption, the zeta potential shifted to -16 mV, this lowering in negative zeta potential reflects electrostatic neutralization as cationic MB^+ ions bind to the MXene surface[51]. This interaction reduces charge density, confirming electrostatic attraction as the primary adsorption mechanism. For $\text{TiO}_2/\text{Ti}_3\text{C}_2$ heterostructures, the initial zeta potential is less negative (-16.9 mV) compared to pristine $\text{Ti}_3\text{C}_2\text{T}_x$ due to absence of ($-\text{Cl}^-$). Post-MB adsorption, the zeta potential further decreases to -11 mV. Comparing the zeta potential values, one can conclude that pristine $\text{Ti}_3\text{C}_2\text{T}_x$ shows better adsorbance properties towards MB removal from waste water, which also makes adsorption an efficient way of MB removal from water rather than photocatalytic dye degradation, which demands more sophisticated materials.

3.2.7 DYE REMOVAL EFFICIENCY

- The MB removal efficiencies of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene and $\text{TiO}_2/\text{Ti}_3\text{C}_2$ heterostructure were examined by conducting dye adsorption test. Real time absorbance vs concentration plots were plotted for both $\text{Ti}_3\text{C}_2\text{T}_x$ MXene and $\text{TiO}_2/\text{Ti}_3\text{C}_2$ heterostructure. The result shows $\text{Ti}_3\text{C}_2\text{T}_x$ removes 92.6% of MB from the stock solution within just 15 minutes and the optimum removal efficiency is 99.42% after 90 minutes, which also reflect in decolorization of the stock solution. Whereas, in the case of $\text{TiO}_2/\text{Ti}_3\text{C}_2$, no significant decolorization is observed and the MB removal efficiency was calculated to be 81.4% only in 90 minutes.

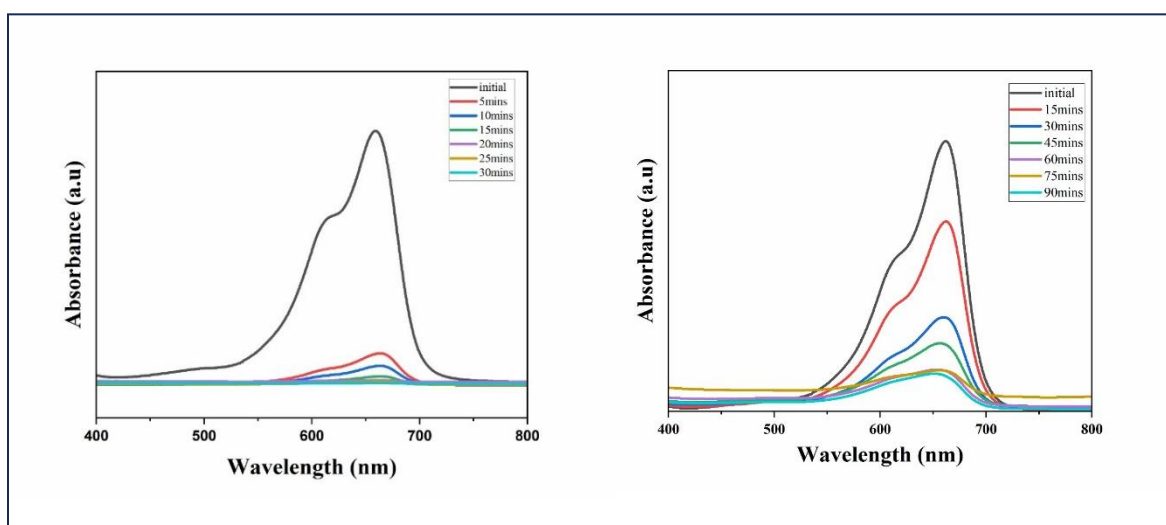


Fig. 14 Real time UV-Vis absorbance plot for (a) $\text{Ti}_3\text{C}_2\text{T}_x$ (b) $\text{TiO}_2/\text{Ti}_3\text{C}_2$

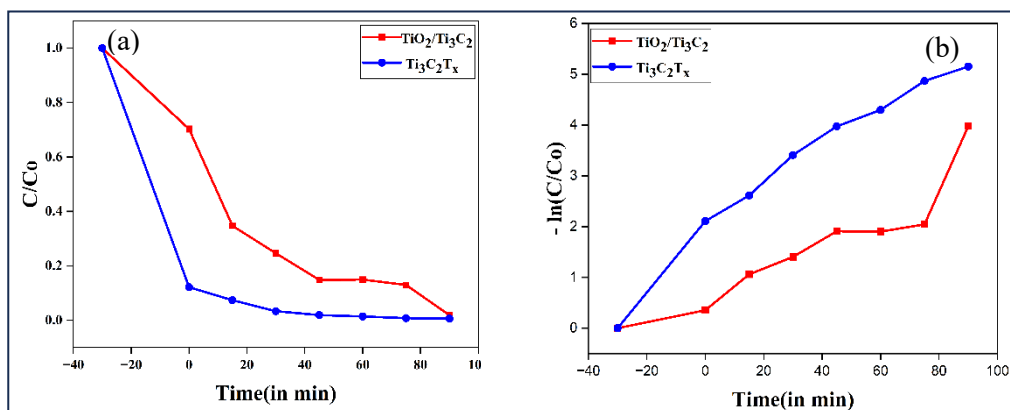


Fig. 15 (a) MB concentration with time (b) Mb removal efficiency with time

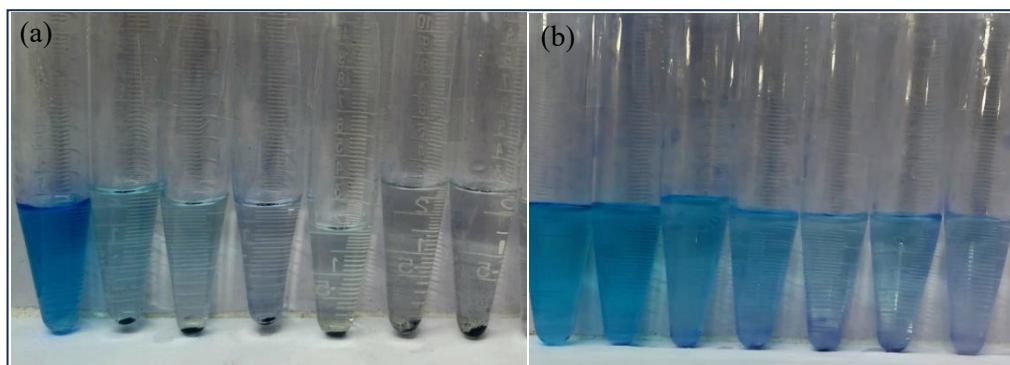


Fig. 16 Decolorization of MB solution using (a) $\text{Ti}_3\text{C}_2\text{T}_x$ (b) $\text{TiO}_2/\text{Ti}_3\text{C}_2$

CHAPTER - 4

CONCLUSIONS AND FUTURE SCOPE

4.1. Conclusion

The synthesized $\text{Ti}_3\text{C}_2\text{T}_x$ MXene demonstrated exceptional methylene blue (MB) adsorption efficiency, achieving 99.42% removal within 90 minutes, attributed to its high specific surface area ($153.63 \text{ m}^2/\text{g}$), negatively charged surface (-31.8 mV zeta potential), and layered structure facilitating electrostatic interactions with cationic dyes. Characterization via XRD confirmed the successful etching of the Al layer from the Ti_3AlC_2 MAX phase, evidenced by the shift of the (002) peak from 9.5° to 6.39° and the disappearance of the (104) peak at 39.1° . FESEM and TEM revealed the transformation of the MAX phase into multilayered MXene nanosheets, while BET analysis highlighted the material's mesoporous nature, crucial for rapid dye uptake. In contrast, the $\text{TiO}_2/\text{Ti}_3\text{C}_2$ heterostructure exhibited limited adsorption (64.6%), likely due to reduced surface terminations and altered charge distribution, as indicated by zeta potential measurements shifting from -16.9 mV to -11 mV post-adsorption. However, the heterostructure's 3D safflower-like morphology, observed in FESEM, and the presence of anatase TiO_2 (confirmed by XRD peaks at 25.3° and 48.4°) suggest untapped potential in dye adsorption.

4.2. Future Scope

Despite their promise, challenges like oxidative instability in humid environments and high production costs persist. Future research focuses on fluoride-free synthesis, surface group optimization, and pilot-scale applications. MXenes bridge material science and environmental engineering, offering a sustainable pathway to mitigate dye pollution. By leveraging their unique properties, MXenes herald a cleaner future for global water resources, demonstrating unparalleled potential in eco-friendly wastewater treatment.

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