

Chitosan/MXene/Polymer Hybrid as a new Filter with Enhanced Hydrophobicity and degradation Properties

Roya Eidi

Ilam University

Ensieh Ghasemian Iemraski

e.ghasemian@ilam.ac.ir

Ilam University

Zohreh Tahmasebi

Ilam University

Article

Keywords: Filter paper, hydrophobic effect, Coating, PVDF, MXene, Chitosan

Posted Date: September 15th, 2026

DOI: <https://doi.org/10.21203/rs.3.rs-10750408/v1>

License:   This work is licensed under a Creative Commons Attribution 4.0 International License.

[Read Full License](#)

Additional Declarations: No competing interests reported.

Chitosan/MXene/Polymer Hybrid as a new Filter with Enhanced Hydrophobicity and degradation Properties

Roya Eidi: Faculty of Science, Department of Chemistry, Ilam University, Ilam, Iran, E-mail: RoyaAidi20257@gmail.com.

Ensieh Ghasemian Lemraski*: Faculty of Science, Department of Chemistry, Ilam University, Ilam, Iran, Corresponding author: E-mail: e.ghasemian@ilam.ac.ir, e.ghasemian@gmail.com, Fax: ++98-843-2227022.

Zohreh Tahmasebi: Faculty of Science, Department of Chemistry, Ilam University, Ilam, Iran. E-mail: z.tahmasebi20@gmail.com.

Abstract

The main objective of this study is to fabricate a hydrophobic filter based on MXene nanosheets and the hydrophobic polymers (PVDF and PDMS), to reduce COD of wastewater. In the first step the MXene nanosheets stabilized using chitosan physically. The formation of a completely homogeneous and uniform film confirms the full coating of the MXene nanosheets with chitosan. The second part of the study involves converting the hydrophilic surface of cellulose filter paper into a hydrophobic surface using a dip-coating method in a PDMS/PVDF solution at different times and ratios. The effect of the polymer ratio and immersion time on the hydrophobicity of the filter paper surface was investigated by measuring the contact angle. Contact angle measurements for all samples showed that the best hydrophobicity was achieved with the filter paper immersed in a polymer solution with a 4:1 ratio for 65 minutes. In the final step, the MXene/chitosan film with uniform thickness was applied onto the optimized filter paper. Structural and morphological characterization techniques were carried out using FTIR, SEM/EDS/MAP, DRS, and XRD, and the results were analyzed. The reduction of COD in water clearly confirms the successful fabrication of the composite filter based on MXene/polymer/filter paper. The main advantage of this filter is the use of low-cost filter paper as a substrate, which is transformed into a hydrophobic filter paper in the absence of commercially available hydrophobic substrates.

Keywords: Filter paper, hydrophobic effect, Coating, PVDF, MXene, Chitosan.

Introduction

Modifying surface hydrophobicity or hydrophilicity is important because it controls how a material interacts with water and other liquids. By adjusting this property, the performance of processes such as water treatment and oil–water separation can be improved, self-cleaning behavior can be achieved, coating adhesion can be enhanced, and corrosion resistance can be increased. Therefore, controlling surface wettability is a key factor in the design of advanced materials. Different methods and techniques can be changed surface energy. Solvent and polymer treatment [1], plasma treatment [2], chemical grafting/surface functionalization [3-6], and heat treatment with hydrophilic additives [7,8] are the most important methods to surface energy modification. Improving surface hydrophobicity is a very common strategy in coatings, membranes, packaging, and biomedical materials. The goal is to reduce surface energy or increase surface roughness, which enhances water repellency (higher contact angle) [9]. Polymers play a crucial role in tailoring surface properties, particularly surface energy and wettability, for a wide range of applications. Materials such as polydimethylsiloxane (PDMS), polyvinylidene fluoride (PVDF), and polytetrafluoroethylene (PTFE) are characterized by intrinsically low surface energy due to the presence of nonpolar methyl groups and highly electronegative fluorine atoms, which promote hydrophobic behavior and water repellency. In contrast, hydrophilic polymers like polyvinyl alcohol (PVA) and chitosan contain abundant polar functional groups (e.g., hydroxyl and amino groups) that enhance surface energy and facilitate strong interactions with water molecules. By selecting appropriate polymers or combining them through blending, coating, or surface modification techniques, it is possible to precisely tune the surface wettability from hydrophilic to hydrophobic regimes. This strategy is particularly important in advanced applications such as coatings, membranes, biomedical devices, and MXene-based composite

films, where interfacial interactions, adhesion, and fluid transport properties are critically dependent on surface energy [10-12].

In this study, a three-layer filtration system was designed and developed for the reduction of chemical oxygen demand (COD) in water. The active layer consists of MXene nanosheets functionalized with chitosan, aimed at enhancing adsorption capacity and improving surface interactions with organic contaminants. The intermediate layer is composed of a PVDF/PDMS polymer blend, which provides a tunable porous structure and enables control over hydrophilic–hydrophobic balance. A cellulose filter paper was employed as the bottom substrate, serving as a mechanical support and ensuring uniform flow distribution across the filter. This hierarchical multilayer configuration was investigated to improve filtration efficiency and enhance the removal performance of organic pollutants from aqueous media [13]. The inherently hydrophilic cellulose substrate was successfully modified into a hydrophobic surface via a simple dip-coating approach, eliminating the need for expensive commercial hydrophobic membranes. The effects of polymer composition and processing conditions on surface properties were systematically investigated, and the fabricated composite filter was thoroughly characterized using advanced analytical techniques. Overall, this study provides a facile and scalable strategy for designing high-performance, low-cost filtration systems with improved efficiency for wastewater treatment applications.

Materials and method

The precursor Ti_3AlC_2 powder (purity >98 wt%) was purchased from Tabibshimi Co., Ltd. Hydrofluoric acid (HF , ≥ 47 wt%), polyvinylidene fluoride (PVDF, $M_w \sim 534,000$ g/mol), chitosan (degree of deacetylation $\sim 85\%$), N,N-dimethylformamide (DMF, $\geq 99\%$), tetrahydrofuran (THF, $\geq 99\%$),

hydrochloric acid (HCl, 37 wt%), and sodium hydroxide (NaOH, $\geq 98\%$) were obtained from Merck (Germany). Polydimethylsiloxane (PDMS), a silicone-based polymer known for its hydrophobic properties, was procured from Behsa Company (Arak, Iran). Cellulose filter paper, typically hydrophilic due to the abundance of hydroxyl groups on cellulose fibers, was supplied by Whatman Company. Deionized water was used throughout all experiments. All chemicals and materials were used as received without further purification.

Loading of PDMS/PVDF mixtures on filter paper

To modify the surface hydrophobicity of the cellulose filter paper, mixture of tetrahydrofuran (THF) and N,N-dimethylformamide (DMF) were prepared in the specified ratios. Then determined amounts of PDMS and PVDF based on Table 1 were added to the solutions and stirred continuously overnight (~ 12 hours) to obtain a completely uniform and homogeneous solution. The solutions were then left undisturbed for 12 hours to allow any bubbles disappeared. Filter papers were subsequently immersed in these polymer solutions in the different time. Then, the filter immersed in distilled water using a reverse-bath method for 24 hours to remove any residual solvents and unbound polymer. Finally, the filter papers were removed and dried again at room temperature for an additional 24 hours. The total of 20 samples were prepared using this method, and the water contact angle of each sample was measured to assess surface hydrophobicity.

Table 1. The solvent ratio, polymer percentage, and immersion time

Sample	THF(mL)	DMF(mL)	PDMS(g)	PVDF(g)	Time(min)
A ₁ -A ₅	5	15	1	0.5	35,50,65,80 & 24 h
B ₁ -B ₅	5	15	2	0.5	35,50,65,80 & 24 h
C ₁ -C ₅	5	15	3	0.5	35,50,65,80 & 24h
D ₁ -D ₅	5	15	4	0.5	35,50,65,80 & 24h

*The samples A-D represent different samples with varying percentages of PVDF/PDMS and time

Loading of MXene film on the filter paper

A 7% hydrofluoric acid (HF) solution in water was prepared. This solution was used as an etchant to selectively remove aluminum from the MAX phase, yielding MXene. A specific amount of MAX phase was mixed with 10 mL of HF7% and stirred for 24 hours. Stirring would facilitate the etching process and helps in obtaining a homogeneous MXene product. The appearance of bubbles during the stirring process indicated proper progression of the etching reaction. This observation suggested that the aluminum layer was being effectively removed from the MAX phase. After the 24-hour stirring the MXene with double distilled water, it was washed to remove residual HF and by-products. Washing continued until the pH of the solution approached neutrality. This ensured that any remaining acidic components were thoroughly removed from the MXene. Finally the remaining residue in the solution was dried in a vacuum at 70 °C for 24 h [14,15]. In the next step, 1 g of chitosan is dissolved in 10 mL of double-distilled water. To facilitate better dissolution of chitosan in water, one drop of acetic acid is added. The solution is then stirred on a magnetic stirrer at room temperature for 3 hours to obtain a uniform and homogeneous solution. Then 0.29 g of MXene dispersed in 5 mL of double-distilled water. The dispersion is sonicated in an ultrasonic bath for 1 hour to ensure proper exfoliation and dispersion of MXene nanosheets [16,17]. After sonication, solutions 1 (chitosan) and 2 (MXene) are combined and sonicated together for 1 h. The mixture is then placed on a magnetic stirrer and

stirred gently for 12 hours at room temperature to obtain a stable and uniform solution with appropriate consistency, suitable for film casting on the filter papers (see Fig. 1). Chemical oxygen demand (COD) was determined using the standard dichromate oxidation method according to established procedures. All measurements were conducted in triplicate, and average values were reported. Fig. 2 shows the preparation of the Chitosan/MXene/Polymer Hybrid and loading on filter paper steps.

Characterization

The microscopic image of the prepared nanocomposite was obtained by Field Emission Scanning Electron Microscopy ((FESEM, QUANTA FEG-450, and FEI, USA)). The crystalline structure of nanocomposite was indicated using an X Ray Diffractometer (PW1730, PHILIPS Company, Netherland). Fourier transform infrared (FTIR) spectroscopy was performed by Bruker-Germany VBRTEX70 spectrometer. UV-vis diffuse reflection spectra (DRS) were performed on a UV-vis spectrophotometer (Diffuse reflectance spectroscopy S-4100, SCINCO, South Korea) with BaSO₄ as the reference compound.

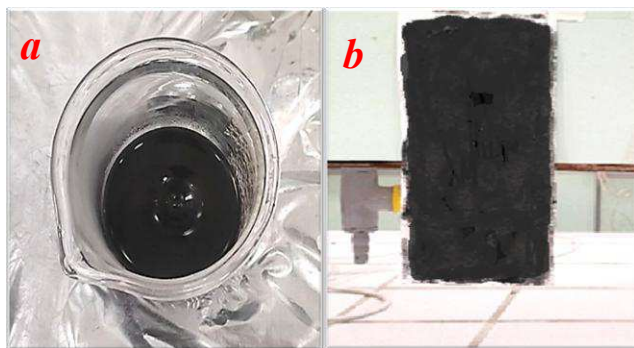


Fig. 1 Homogeneous mixture of (a) MXene and chitosan; (b) chitosan/MXene casting on the filter papers.



Fig. 2 schematic image of preparation processes of modified filter paper

Results & Discussion

In general the diffusion and spreading rate of polymer solution on a substrate surface depends on kind of polymers, solvent, structure of the substrate, concentration of polymer, temperature, method, and time. These factors control the hydrophobicity properties of substrate due to the interfacial interactions. The choice of solvent plays a crucial role in the diffusion and spreading of polymers on a substrate during immersion-based coating or deposition methods (like dip coating, solvent casting, or solution immersion) [16-18]. In the present work the mixture of THF/DMF has been used as a solvent bath for polymers loading on the filter papers at different polymer ratio and immersion time. For all the prepared samples, the measured contact angle reported in Table 2.

Table 2 Contact angle of the modified filter paper at different time and PDMS/PVDF ratio.

Sample	PDMS/PVDF (w/w)	Immersion time (min)	Contact angle (Degree)
A ₁	2	35	41.81
A ₂	2	50	70.43
A ₃	2	65	58.33
A ₄	2	80	85.96
A ₅	2	24 h	81.35
B ₁	4	35	104.15
B ₂	4	50	101.42
B ₃	4	65	120.12
B ₄	4	80	104.13
B ₅	4	24 h	101.42
C ₁	6	35	83.21
C ₂	6	50	85.46
C ₃	6	65	89.67
C ₄	6	80	81.35
C ₅	6	24 h	88.37
D ₁	8	35	48.53
D ₂	8	50	33.78
D ₃	8	65	41.81
D ₄	8	80	48.2
D ₅	8	24 h	46.5

Immersion time as an important parameter impacts the surface morphology, coating thickness, water repellency and ultimately the surface hydrophobicity [19,20]. The reported results in Table 2, and Fig. 3a and 3b confirmed this phenomena. For the PDMS/PVDF: 2(sample A), the contact angle increased with immersion time, reaching a peak at 80 min, and slightly decreased after 24 hours, suggesting enhanced hydrophobicity with increasing immersion time. In the case of PDMS/PVDF: 4(sample B), the contact angle rises steadily up to 65 min, then slightly decrease at 80 min and remains relatively stable through 24 hours, indicating that this ratio may provide an optimal balance between surface roughness and low surface energy. When the ratio of polymers is increased to 6(sample C) , a general increasing trend in contact angle is observed, it shows a peak at 65 minutes, followed by a minor decline, potentially due to surface saturation or polymer rearrangement. Conversely, at PDMS/PVDF: 8(sample D), the contact angle shows a fluctuating pattern without a consistent trend, which may result from excess PDMS

reducing surface roughness or phase separation effects, thus limiting further improvement in hydrophobicity. These trends are consistent with reported studies on polymer blend surface coatings, where moderate PDMS content enhances hydrophobicity through synergistic effects, while excessive PDMS can lead to smoother, less textured surfaces with reduced contact angles [21-24]. Variation of contact angle versus immersion time for all the prepared samples respectively are given in Figs. 3a, and 3b.

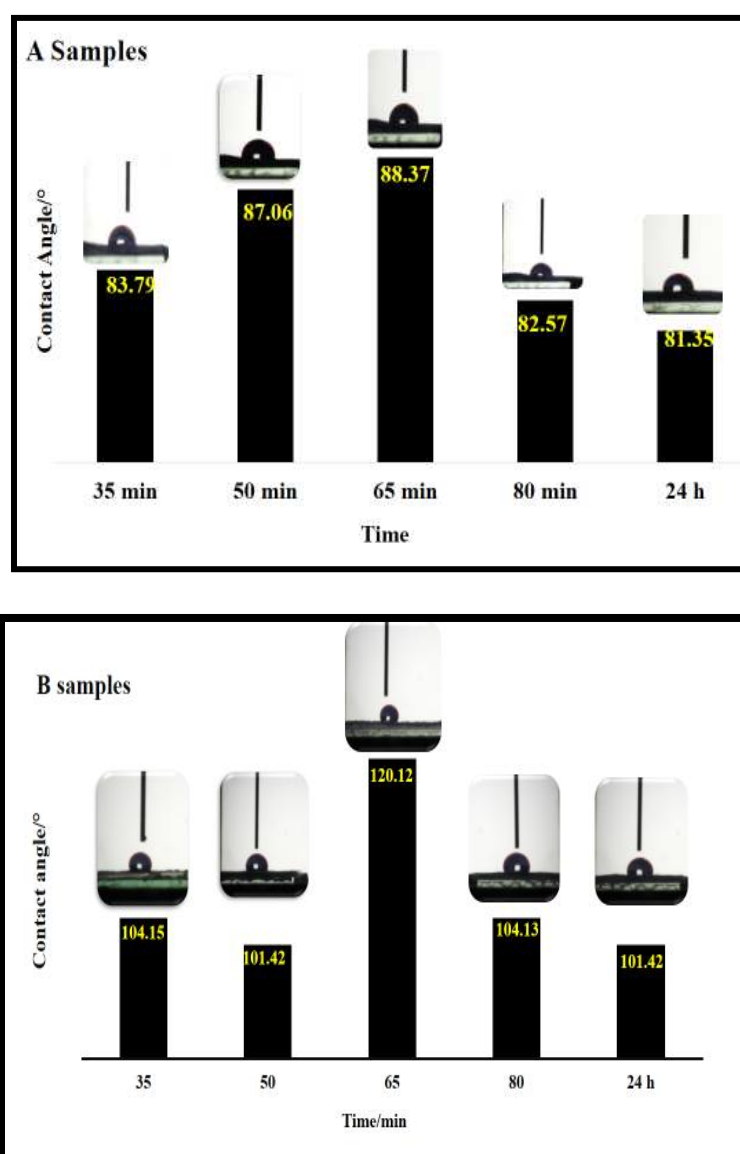


Fig. 3a Snapshots of a water droplet impinging on the hydrophobic samples A, and B.

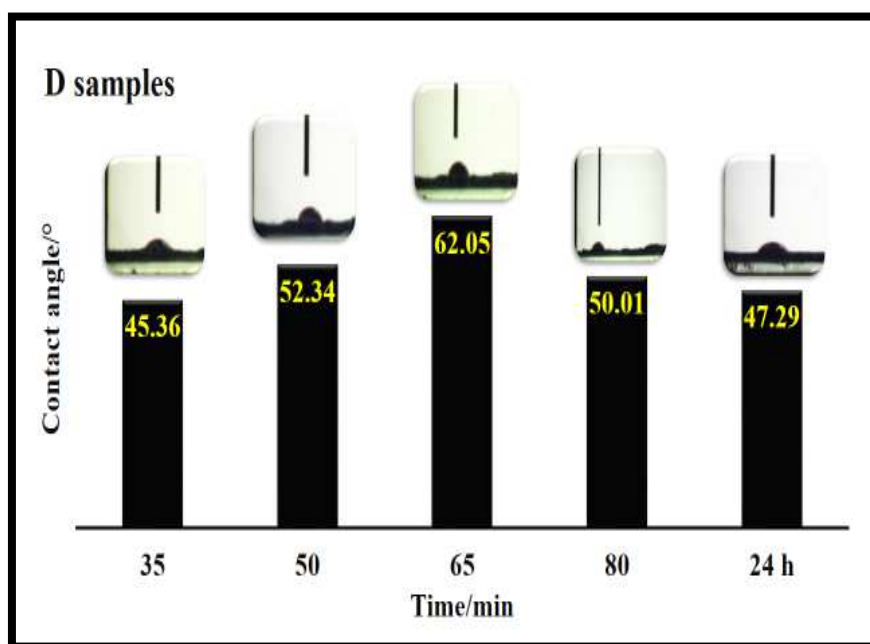
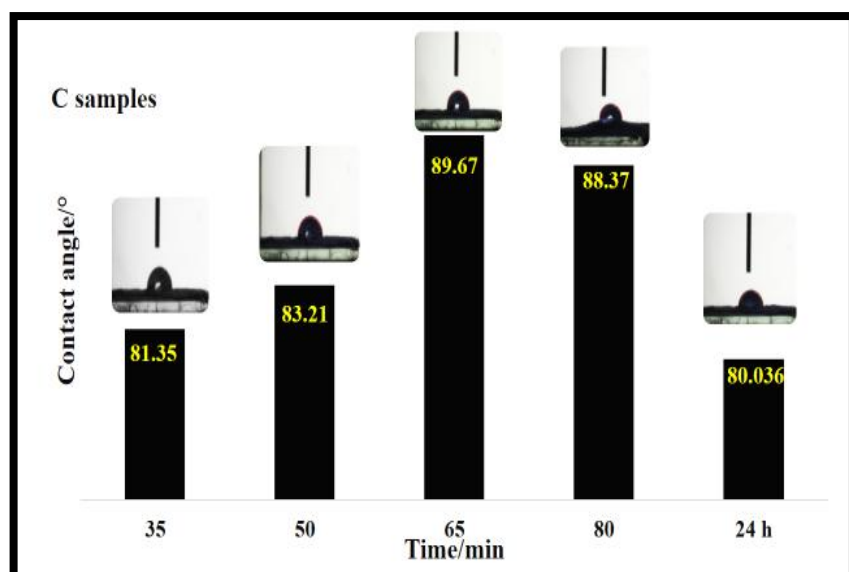


Fig. 3b Snapshots of a water droplet impinging on the hydrophobic samples C, and D.

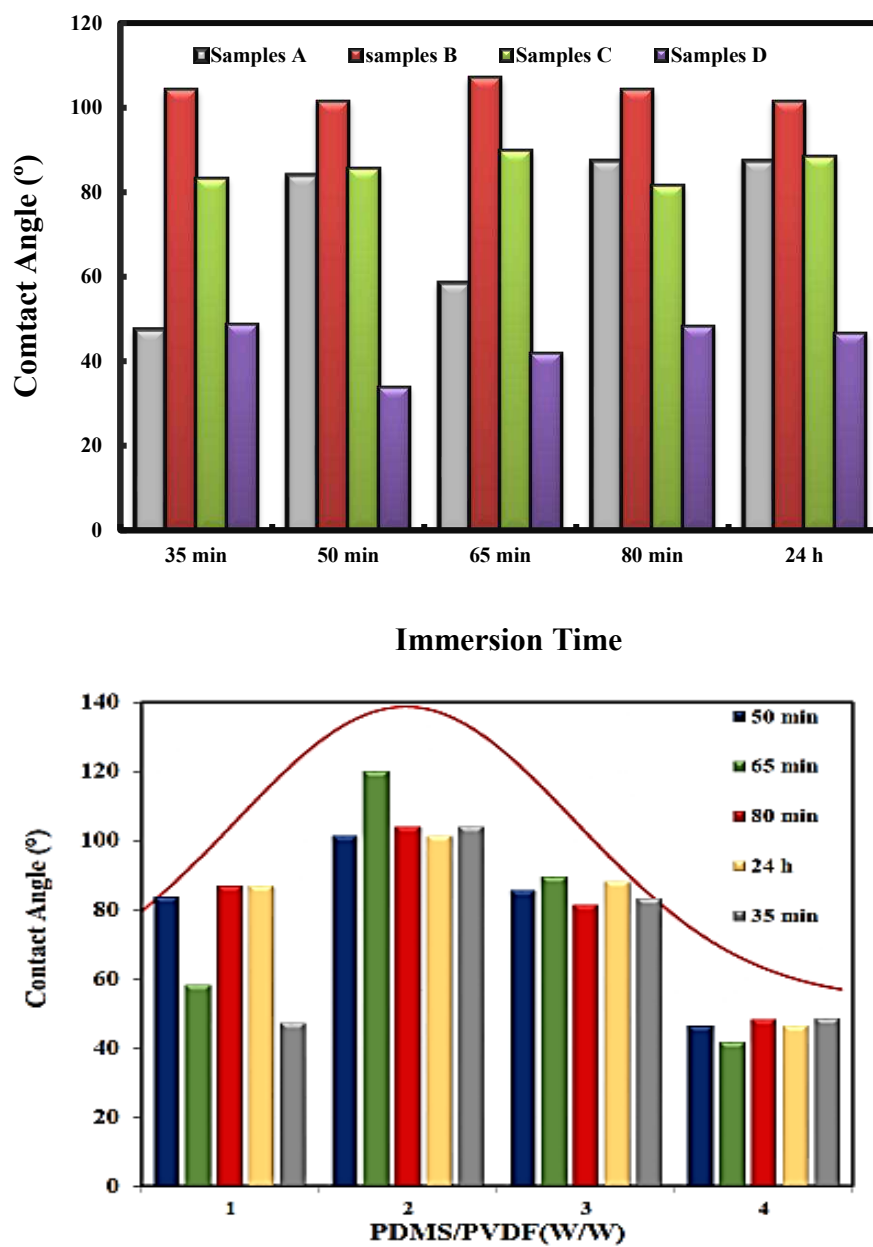


Fig. 4 Variation of contact angle versus (a) immersion time and (b) PDMS/PVDF(W/W) for all the prepared samples.

Another critical parameter in achieving the desired hydrophobic surface is the variation in polymer content, as it governs the diffusion and coverage behavior of the casting solution on the substrate. A thorough comparison of the results presented in Table 2 and Fig. 4B indicates that the contact angle attains its maximum value at a specific ratio of the two polymers. The maximum contact angle at the ratio PDMS/PVDF:4, indicating a more hydrophobic surface. In contrast, the contact angle decreased at the 2 and 8 w/w ratios, reflecting lower hydrophobicity, while the 6 w/w ratio exhibited moderate hydrophobicity with a contact angle value of approximately 83°.

At a low PDMS content (2 w/w), the contact angle is lower than 90°, indicating a relatively hydrophilic surface. This suggests that at low PDMS concentration, the hydrophobic polymer (PDMS) is insufficient to significantly alter the hydrophilic nature of the PVDF-coated filter paper. PVDF (Polyvinylidene fluoride) itself is somewhat hydrophobic, but when mixed at low PDMS content, the overall surface does not become significantly water-repellent. The contact angle peaks sharply at 104.12° at ratio PDMS/PVDF: 4 w/w, indicating a transition to a highly hydrophobic surface. This substantial increase suggests that the addition of PDMS (polydimethylsiloxane), a highly hydrophobic silicone-based polymer, effectively coats or embeds within the surface, creating a rough, low-energy surface that repels water droplets. PDMS is known for its very low surface energy (about 20 mN/m), which promotes hydrophobicity [25-27].

Interestingly, increasing PDMS beyond 4 w/w to 6 and 8 w/w leads to decrease of contact angle respectively. This decline suggests that excess PDMS may negatively affect surface properties, possibly due to phase separation or aggregation. Too much PDMS could lead to microphase separation or non-uniform coating, exposing hydrophilic PVDF regions or creating a less ideal

surface texture. Excess PDMS might fill surface pores or smooth out roughness, reducing the surface roughness and thus decreasing hydrophobicity. On the other hands high PDMS content might cause a rearrangement of molecular chains that exposes more hydrophilic groups or reduces the effectiveness of the hydrophobic coating. These behaviors are consistent with previous studies on polymer blends where an optimal hydrophobic polymer concentration maximizes water repellency, but beyond that, the surface properties deteriorate [28,29].

Characterization of chitosan/MXene

MXene surface typically has termination groups denoted T_x , like $-OH$, $-O$, $-F$, sometimes $-Cl$ etc. The titanium atoms on the surface (Ti sites) can coordinate or form bonds with oxygen, hydroxyl, or other functional groups. On the other hands. Chitosan is a polysaccharide with repeating units of glucosamine (and some N-acetyl glucosamine, hydroxyl ($-OH$) groups (on the sugar ring) and amide linkages (especially if some residual N-acetyl groups). From the literature, the interactions between surface functional group of MXene and chitosan usually are non-covalent, but in some cases possibly some kinds of covalent or strong electrostatic interactions has been occurred. FTIR spectra of chitosan/MXene is given in Fig. 5. The broad absorption band at $\sim 3445\text{ cm}^{-1}$ corresponds to overlapping $O-H$ and $N-H$ stretching vibrations of chitosan and surface $-OH$ groups of MXene, indicating strong hydrogen bonding. The band at $\sim 1632\text{ cm}^{-1}$ is attributed to the amide I vibration of chitosan and interlayer water of MXene. The peaks observed at ~ 787 and $\sim 549\text{ cm}^{-1}$ are characteristic of $Ti-O/Ti-C$ vibrations, confirming the presence of MXene in the composite [29].

XRD patterns of MXene nanosheet in Fig. 6A exhibits several sharp peaks (high crystallinity) e.g. at 18.3° (004), 27.3° (006), 40° (103), 61.2° (110). In the chitosan/MXene

composite major peaks appear at $\sim 10.30^\circ$, $\sim 20.0^\circ$, $\sim 38^\circ$, and $\sim 61.3^\circ$. The MXene peaks shift (typically toward lower angles) in the composite, indicating an increase in d-spacing (i.e. interlayer expansion) due to interaction/intercalation of chitosan molecules between MXene layers. The intensity of MXene crystallographic peaks decreases sharply in the composite, reflecting a loss or reduction of crystallinity, structural distortion, or disruption of regular stacking in MXene due to the presence of chitosan. The distinct chitosan peak at $\sim 20^\circ$ remains visible, sometimes overlapping or shifting slightly [29,30].

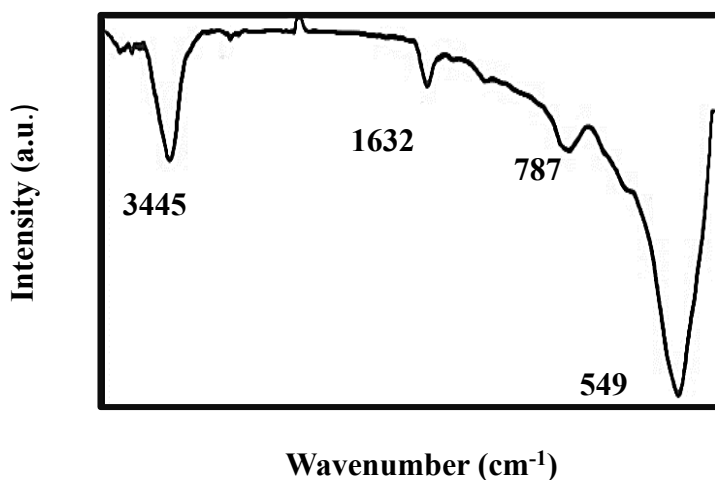


Fig. 5 FTIR spectrum of chitosan loaded on MXene nanosheet

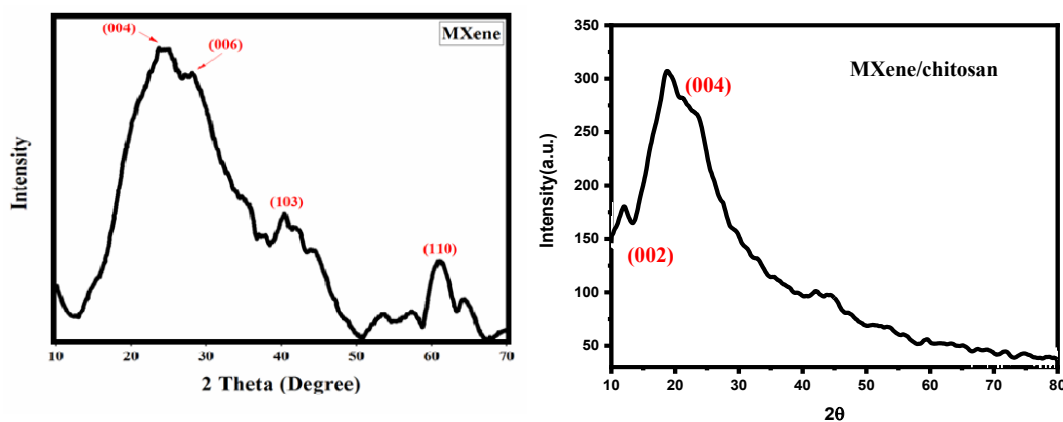


Fig. 6A XRD patterns of MXene, and chitosan loaded on MXene nanosheet

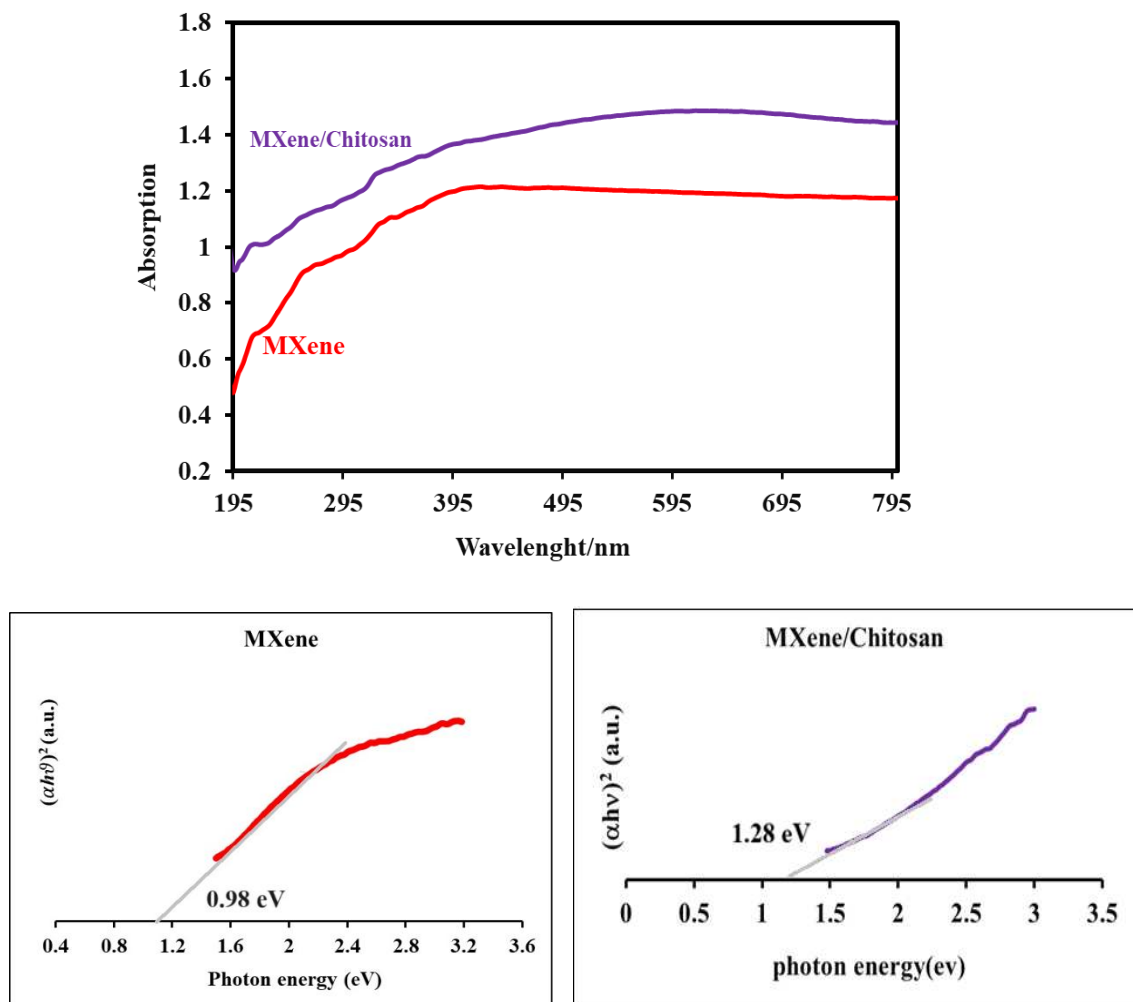


Fig. 6b XRD patterns; DRS graph, and Plot of $(\alpha h\nu)^2$ vs. photon energy of the studied samples

In the DRS (Diffuse Reflectance Spectroscopy) graph in Fig. 6b the MXene shows a relatively stable absorption starting from around 0.5 at 195 nm and gradually increasing toward 1.2. The Chitosan/MXene starts from a higher absorption value of approximately 0.95 and rises sharply to around 1.5 before leveling off. The MXene/Chitosan composite has a higher absorption across the entire wavelength range compared to the MXene alone. This suggests that chitosan may enhance the absorption properties of MXene. Both spectra show a relatively smooth

absorption increase from about 200 nm to 795 nm, with MXene/Chitosan exhibiting a slightly steeper slope, especially in the longer wavelength region (around 600 nm and above). MXene/Chitosan shows a more prominent absorption peak, particularly in the longer wavelengths, indicating that the incorporation of chitosan increases the material's ability to absorb light in that range. This data could suggest that the addition of chitosan to MXene improves its light absorption, possibly due to enhanced electronic or optical properties introduced by the chitosan polymer [30-32]. FESEM image in Fig. 7a shows modified filter paper covered by chitosan/MXene solution. The FESEM images reveal a rough and hierarchical surface morphology consisting of stacked MXene nanosheets embedded in a chitosan matrix on the filter paper. This structure provides a high surface area and partially reduces the effective pore size, enhancing the adsorption and physical retention of organic contaminants responsible for COD. Additionally, the increased surface roughness and flow path tortuosity improve contact between the wastewater and the active filtration layer, leading to efficient COD reduction while maintaining adequate permeability [33]. EDS mapping in Fig. 7b shows distribution of elements e.g. Ti, C, O, N (from MXene and Chitosan) vs F (from PVDF) etc. Homogeneity of Ti map indicates good dispersion; sharp Ti boundary means coating layer distinct; mixing or gradient suggests infiltration or diffusion. Also presence of oxidation (high O/Ti at edges) might suggest degradation. A compact, multilayered coating is clearly observed on the cellulose fiber network, confirming successful deposition and strong interfacial adhesion between the hybrid composite and the filter substrate. The scale bar corresponds to 100 μm .

The cross-sectional SEM image in Fig. 7c reveals a well-defined multilayer architecture formed on the filter paper substrate. A dense and continuous top layer composed of stacked MXene nanosheets embedded within the chitosan/polymer matrix is observed, followed by a gradually

transitioning interfacial region where the hybrid coating infiltrates and anchors into the cellulose fiber network. This hierarchical layer structure ensures strong interfacial adhesion and mechanical stability of the coating.

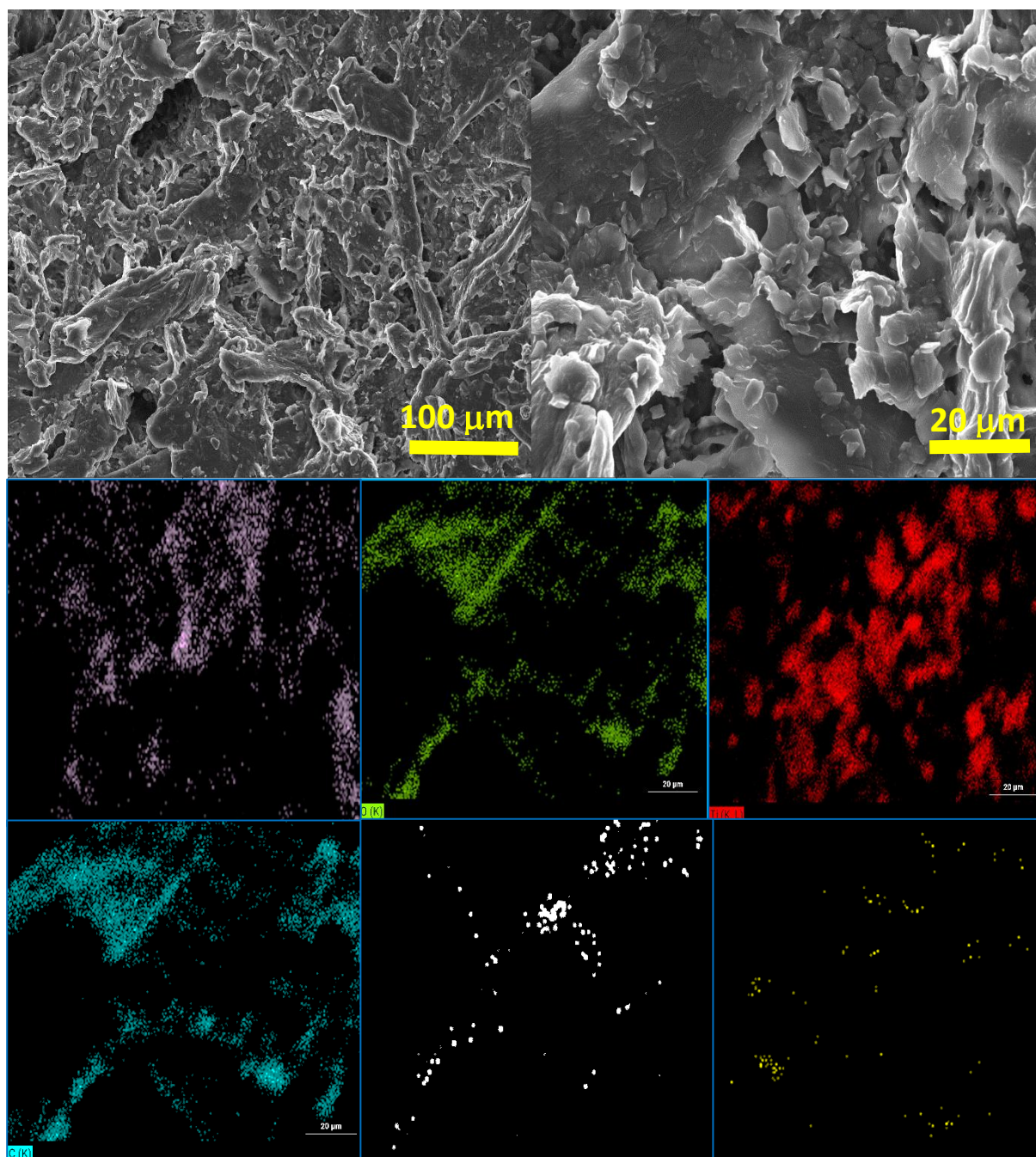


Fig 7a. FESEM, Map of filter paper covered by Chitosan/MXene/Polymer Hybrid

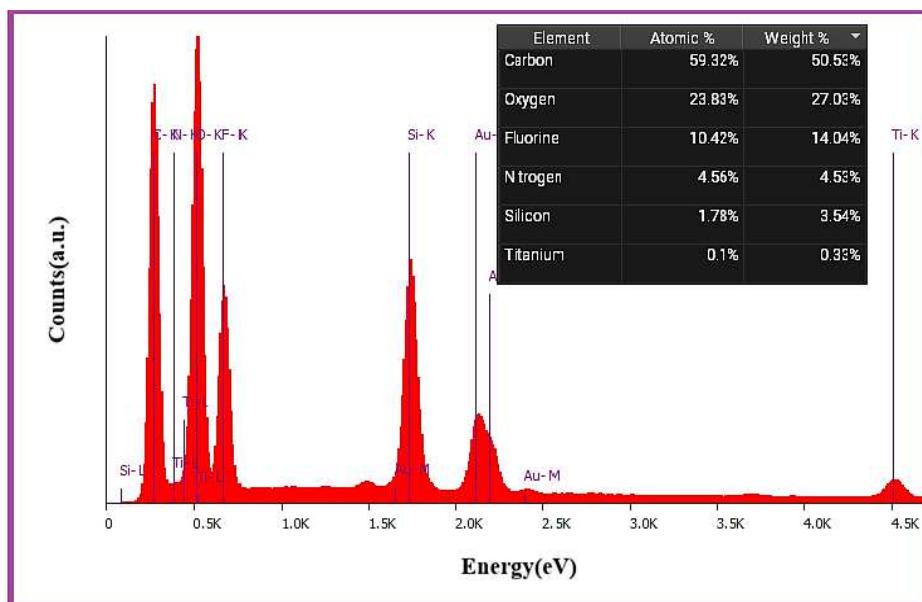
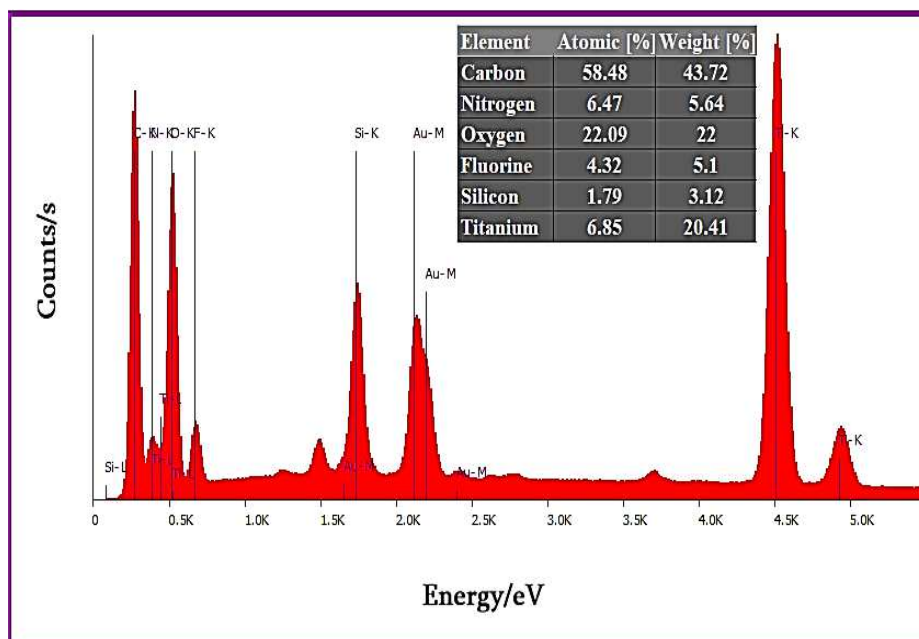


Fig 7b. EDS of filter paper and covered by Chitosan/MXene/Polymer Hybrid

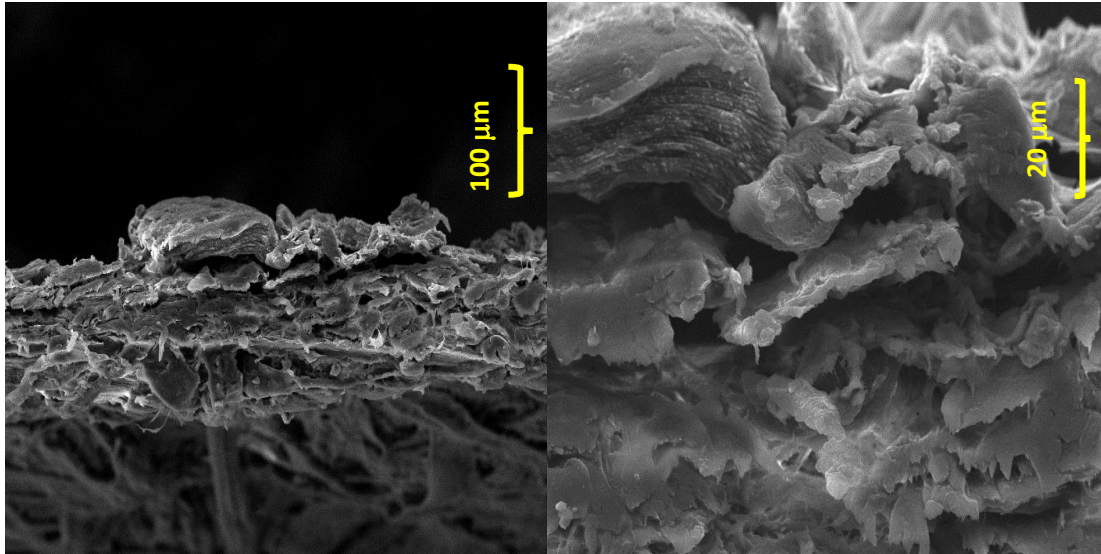


Fig 7c. Cross section of filter paper covered by Chitosan/MXene/Polymer Hybrid

The multilayer architecture of the chitosan/MXene/polymer-coated filter paper plays a decisive role in regulating mass transport and separation efficiency during filtration. The dense and highly stacked MXene nanosheet layer acts as the primary barrier for contaminant rejection. The two-dimensional lamellar structure forms sub-nanometer to nanometer-scale interlayer channels, which effectively restrict the passage of large molecules, colloids, and microorganisms through size exclusion. In addition, the negatively charged surface terminations of MXene ($-O$, $-OH$, $-F$) promote electrostatic interactions and adsorption of positively charged species, such as heavy metal ions (e.g., Pb^{2+} , Cu^{2+}) and cationic dyes, enhancing contaminant retention at the membrane surface. The intermediate layer serves as a transport-regulating and stabilization zone. The chitosan/polymer matrix intercalates between MXene sheets, preventing excessive restacking and maintaining controlled nanochannels for molecular transport. Chitosan provides abundant amino and hydroxyl functional groups, enabling chelation and hydrogen bonding with metal ions and polar organic molecules. This layer allows selective permeation of water molecules while slowing

down contaminant diffusion via tortuosity increase and affinity-based interactions. The porous cellulose fiber network functions as a mechanical support and flow distributor, minimizing pressure drop and ensuring uniform permeation. Partial infiltration of the hybrid coating into the cellulose matrix creates a graded interface, which enhances interfacial adhesion and suppresses delamination during filtration. This open macroporous structure facilitates rapid transport of purified water while preventing back-diffusion of retained contaminants.

COD Results

The effects of irradiation time, pH, and H₂O₂ concentration on COD reduction using prepared filter are given in Table 3. All of obtained data confirmed the COD values at acidic pH decreased via increasing contact time from 20 to 120 min, corresponding to a ~59% COD removal.

Table 3. Obtained COD percent at different conditions (T=298.15 K, Initial COD:800 ppm)

Sample	PH	Contact time/min	H ₂ O ₂ /ppm	COD	R%
1	4	20	-	773	3.375
2	4	40	-	631	21.125
3	4	60	-	602	24.75
4	4	80	-	388	51.5
5	4	100	-	327	59.125
6	4	120	-	321	59.875
1	10	20	-	790	1.25
2	10	40	-	475	40.625
3	10	60	-	350	56.25
4	10	80	-	310	61.25
5	10	100	-	302	62.25
6	10	120	-	300	62.5
1	7	120	20	740	7.5
2	7	120	30	719	10.125
3	7	120	40	577	27.875
4	7	120	50	451	43.625

At acidic pH rapid COD reduction is observed up to 80 min and then approaches a plateau around 100-120 min. Oxidative reactions are occurred more due to the enhanced formation of reactive oxygen species (ROS). The plateau at longer irradiation times indicates depletion of readily oxidizable organic compounds. On the other hands at basic pH, COD decreased over 20–120 min, corresponding to a ~62.0% COD removal, slightly higher than pH 4. Alkaline conditions favor hydroxyl radical formation (via OH^- oxidation), improving mineralization efficiency. Higher COD removal at alkaline pH suggests improved oxidative degradation due to increased $\bullet\text{OH}$ availability and better charge interactions between pollutants and reactive species.

At pH 7 and constant irradiation time (120 min), increasing H_2O_2 concentration results in a pronounced decrease in COD. Increasing H_2O_2 concentration significantly enhances COD removal due to increased generation of hydroxyl radicals ($\bullet\text{OH}$). The strong downward trend indicates that the system operates in the radical-limited regime within this concentration range. Since no plateau is observed up to 50 ppm, the optimal H_2O_2 concentration has not yet been reached, and higher dosages may further improve COD reduction until scavenging effects appear.

[34]

The reduction in chemical oxygen demand (COD) observed for the fabricated photomembrane is primarily attributed to the MXene-rich functional layer integrated within the polymer-based membrane structure. Under light irradiation, MXene nanosheets act as the main active component, facilitating the generation of reactive oxygen species that oxidize organic contaminants, thereby contributing directly to COD reduction. In parallel, the high surface area and layered morphology of MXene provide effective adsorption sites, promoting the accumulation of organic molecules at the reactive interface and enhancing degradation efficiency.

Chitosan plays a critical supporting role by serving as a polymeric interlayer that bridges the hydrophobic PVDF/PDMS-modified filter paper and the MXene nanosheets, preventing nanosheet restacking and preserving accessible active sites while improving mass transport through electrostatic interactions. The PVDF/PDMS coating transforms the inherently hydrophilic cellulose filter paper into a hydrophobic substrate, regulating water permeation, increasing residence time, and improving the mechanical stability of the membrane during operation. The cellulose fiber network itself primarily functions as a porous support, enabling continuous flow without directly participating in pollutant degradation. The synergistic combination of MXene photocatalytic activity and polymer-mediated structural control results in a multifunctional photomembrane capable of simultaneous filtration and oxidative degradation, leading to enhanced COD removal performance [34].

Conclusion

In this study, a hydrophobic polymer–ceramic-based filter was successfully developed through a surface-engineering strategy. Hydrophobic modification of commercial filter paper using PVDF/PDMS coatings was systematically optimized by controlling polymer composition and immersion time. Contact angle analysis demonstrated that both parameters play a critical role in determining surface wettability and hydrophobicity. This optimization enabled a reliable change of the hydrophilic filter paper substrate into a robust hydrophobic surface. The fabricated chitosan/MXene/polymer hybrid uniformly deposited onto a cellulose filter paper substrate. MXene nanosheets were subsequently covered with chitosan to enhance its interfacial compatibility and stability. The resulting multilayer filter comprises a dense MXene-rich top layer acting as a degradation agent and adsorption barrier, an intermediate chitosan/polymer matrix regulating mass transfer and stabilizing MXene interlayer spacing, and a porous cellulose fiber

network providing mechanical support and flow distribution. The MXene-based layer facilitates the generation of reactive oxygen species, while the membrane structure enables simultaneous degradation and physical separation of organic contaminants, leading to enhanced COD removal efficiency [34].

Acknowledgements

The authors are grateful for the financial support of Ilam University.

Funding Declaration

This research received no specific grant from any funding agency in the public, commercial, or not-for-profit sectors.

Author Contribution

Roya Eidi: Investigation, Data curation, Methodology, and Formal analysis,. Ensieh Ghasemian Lemraski: Conceptualization, Methodology, Supervision, Validation, Writing – review and editing, and Project administration. Zohreh Tahmasebi: Investigation, Data curation, Formal analysis, and Writing – review and editing. All authors have read and approved the final version of the manuscript.

Disclosure

The authors report no potential conflicts of interest for this work.

References

1. Li, J., Z. Liu, R. Zhang, Luo, G., Sun, Y., Zhang, J., Shen Q. “Surface Treatment and Solvent Co-Assisted Easy Direct Bonding of Polymer/Metal,” *Materials & Design* 204 (2021): 109641. <https://doi.org/10.1016/j.matdes.2021.109641>.
2. Giraldo-Mejía, H., Y. M. Quintero, F. Mery, et al., “Plasma-Grafting Surface Modifications to Enhance Membrane Hydrophobicity for Brine Membrane Distillation,” *Desalination* 567 (2023): 116942. <https://doi.org/10.1016/j.desal.2023.116942>.
3. Bouiahya, K., A. Oulguidoum, A. Laghzizil, et al., “Hydrophobic Chemical Surface Functionalization of Hydroxyapatite Nanoparticles for Naphthalene Removal,” *Colloids and Surfaces A: Physicochemical and Engineering Aspects* 595 (2020): 124706. <https://doi.org/10.1016/j.colsurfa.2020.124706>.
4. Nuo, Xu., Guangyu, Pan., Hui, Zhang., Peng, Lu., Lei, Shen., Yuguang, Li., Dong, Ji., Jindian, D., Xin, H., Chunhua, L., Zhongzi, X., Ning, Z., Kai, G., PVDF-Based Fluoropolymer Modifications via Photoinduced Atom Transfer Radical Polymerizations, *Advances in Polymer Technology*, (2022) 7798967, <https://doi.org/10.1155/2022/7798967>.
5. Zhang, Y., Q. Liu, J. Li, et al., “Large-Scale Fabrication of Flexible Antiglare Film with Superhydrophobic Surface toward High Transmittance and High Haze,” *Chemical Engineering Journal* 511 (2025): 161805. <https://doi.org/10.1016/j.cej.2025.161805>.
6. Qi, X., J. Wu, L. He, et al., “Silane-Grafted Graphene Superhydrophobic Coating on Nonwoven Fabric for Photothermal-Driven High-Viscosity Oil–Water Separation,” *Carbon* 233 (2025): 119886. <https://doi.org/10.1016/j.carbon.2024.119886>.

7. Sun, Y., S. Sun, X. Liao, et al., “Effect of Heat Treatment on Surface Hydrophilicity-Retaining Ability of Titanium Dioxide Nanotubes,” *Applied Surface Science* 440 (2018): 440–447. <https://doi.org/10.1016/j.apsusc.2018.01.136>.
8. Ngo, H. V., P. K. Nguyen, T. V. Vo, et al., “Hydrophilic–Hydrophobic Polymer Blend for Modulation of Crystalline Changes and Molecular Interactions in Solid Dispersion,” *International Journal of Pharmaceutics* 513 (2016): 148–152. <https://doi.org/10.1016/j.ijpharm.2016.09.017>.
9. Tian, C., J. Chen, X. Li, et al., “Chemical Cleaning–Solvent Treatment–Hydrophilic Modification Strategy for Regenerating End-of-Life PVDF Membrane,” *Journal of Membrane Science* 669 (2023): 121325. <https://doi.org/10.1016/j.memsci.2022.121325>.
10. Vásquez, L., A. Davis, F. Gatto, et al., “Multifunctional PDMS PolyHIPE Filters for Oil–Water Separation and Antibacterial Activity,” *Separation and Purification Technology* 255 (2021): 117748. <https://doi.org/10.1016/j.seppur.2020.117748>.
11. Zhang, W., Y. Shi, B. Wang, et al., “High-Strength Electrospun Polydimethylsiloxane/Polytetrafluoroethylene Hybrid Membranes with Stable and Controllable Coral-Like Structures,” *Composites Part A: Applied Science and Manufacturing* 164 (2023): 107316. <https://doi.org/10.1016/j.compositesa.2022.107316>.
12. Abbasi Moud, A., “Polymer-Based Flocculants: Review of Water Purification Applications,” *Journal of Water Process Engineering* 48 (2022): 102938. <https://doi.org/10.1016/j.jwpe.2022.102938>.
13. Rana, A. K., V. K. Gupta, P. Hart, et al., “Sustainable MXene–Chitosan/Chitin Composites for Interdisciplinary Applications,” *Materials Today Sustainability* 25 (2024): 100671. <https://doi.org/10.1016/j.mtsust.2024.100671>.

14. Tahmasebi, Z., and E. Ghasemian Lemraski, “Ternary Zinc Oxide–Tungsten Oxide–MXene Nanocomposite for Photocatalytic and Photoelectrocatalytic Desulfurization,” *Electrochemistry Communications* 177 (2025): 107933. <https://doi.org/10.1016/j.elecom.2025.107933>.
15. Naguib, M., M. Kurtoglu, V. Presser, et al., “Two-Dimensional Nanocrystals Produced by Exfoliation of Ti_3AlC_2 ,” *Advanced Materials* 23 (2011): 4248–4253. <https://doi.org/10.1002/adma.201102306>.
16. Majerczak, K., Z. Shi, Z. Zhang, et al., “Diffusion Kinetics of Molecular Probe in Thin Poly(vinyl alcohol)-Based Films,” *Progress in Organic Coatings* 185 (2023): 107833. <https://doi.org/10.1016/j.porgcoat.2023.107833>.
17. Erdem, R., İ. Usta, M. Akalin, et al., “Impact of Solvent Type and Mixing Ratios on Polyurethane Electrospun Nanofibers,” *Applied Surface Science* 334 (2015): 227–230. <https://doi.org/10.1016/j.apsusc.2014.10.123>.
18. Lee, K. H., H. Y. Kim, Y. M. La, et al., “Influence of Mixing Solvent on Electrospun Poly(vinyl chloride) Nonwoven Mats,” *Journal of Polymer Science Part B: Polymer Physics* 40 (2002): 2259–2268. <https://doi.org/10.1002/polb.10293>.
19. Shi, X., L. Wang, N. Yan, et al., “Fast Evaporation-Enabled Ultrathin Polymer Coatings on Nanoporous Substrates for Highly Permeable Membranes,” *The Innovation* 2 (2021): 100088. <https://doi.org/10.1016/j.xinn.2021.100088>.
20. Al-Hajri, S., S. M. Mahmood, H. Abdulelah, et al., “An Overview on Polymer Retention in Porous Media,” *Energies* 11 (2018): 2751. <https://doi.org/10.3390/en11102751>.

21. Sun, D., M.-Q. Liu, J.-H. Guo, et al., “Preparation and Characterization of PDMS–PVDF Hydrophobic Microporous Membrane for Membrane Distillation,” *Desalination* 370 (2015): 63–71. <https://doi.org/10.1016/j.desal.2015.05.017>.
22. Zhang, B., P. W. Wong, J. Guo, et al., “Transforming $\text{Ti}_3\text{C}_2\text{T}_x$ MXene Intrinsic Hydrophilicity into Superhydrophobicity for Efficient Photothermal Membrane Desalination,” *Nature Communications* 13 (2022): 3315. <https://doi.org/10.1038/s41467-022-31028-6>.
23. Pyo, M., K. H. Lee, and E.-J. Lee, “Enhancing Hydrophobicity through Polydimethylsiloxane-Blended Poly(vinylidene fluoride-co-hexafluoropropylene) Electrospun Membranes,” *Polymer* 307 (2024): 127261. <https://doi.org/10.1016/j.polymer.2024.127261>.
24. Rioboo, R., I. Demnati, M. Amin Ali, et al., “Superhydrophobicity of Composite Surfaces Created from Polymer Blends,” *Journal of Colloid and Interface Science* 555 (2019): 707–715. <https://doi.org/10.1016/j.jcis.2019.10.043>.
25. Montero Rocca, F. E., et al., “Hydrophobic PVDF-Based Electrospun Membranes: Design Criteria, Fabrication, and Resistance to Long-Term Hydrodynamic Operation,” *Journal of Applied Polymer Science* (2025): e57577. <https://doi.org/10.1002/app.57577>.
26. Liu, J., Z. Cai, and Y. Ji, “Synthesis of Bio-Based Modified Amphoteric Epoxy Acrylate Sizing Emulsion and Its Application in Paper Sizing,” *Journal of Applied Polymer Science* (2023): e54462. <https://doi.org/10.1002/app.54462>.
27. Ghasemian Lemraski, E., S. Alibeigi, and Z. Abbasi, “Ibuprofen@Silver-Loaded Poly(vinyl alcohol)/Chitosan Copolymer Scaffold as a Novel Drug Delivery System,” *Materials Today Communications* 31 (2022): 103640.

28. Ghasemian Lemraski, E., H. Jahangirian, M. Dashti, et al., “Antimicrobial Double-Layer Wound Dressing Based on Chitosan/Poly(vinyl alcohol)/Copper: In Vitro and In Vivo Assessment,” *International Journal of Nanomedicine* 13 (2018): 223–235.
29. Doğan Ulu, Ö., A. Ulu, E. Birhanlı, et al., “Fabrication and Characterization of Functional Chitosan/MXene Composite Films Decorated with Lead Zirconate Titanate Nanoparticles,” *Materials Chemistry and Physics* 343 (2025): 131022. <https://doi.org/10.1016/j.matchemphys.2025.131022>.
30. Elbialy, N. S., S. F. Aboushoushah, N. Mohamed, et al., “Chitosan-MXene-Silver Nanocomposite Film with Improved Physicochemical and Biological Functionalities to Potentiate Wound Healing Process,” *Journal of Drug Delivery Science and Technology* 111 (2025): 107133. <https://doi.org/10.1016/j.jddst.2025.107133>.
31. Qin, Y., K. Su, Y. Zhao, et al., “Chitosan-Integrated MXene-Based Nanofiltration Membrane With High Permeability for Efficient Dye Wastewater Treatment,” *Journal of Applied Polymer Science* 142 (2025): e57157. <https://doi.org/10.1002/app.57157>.
32. Khan, A. A., M. Tahir, A. Sherryana, et al., “Synthesis and Characterization of TiC MXene-Based Composites for Energy Storage and Conversion,” in *Titanium Carbide MXenes*, ed. M. Tahir (2024). <https://doi.org/10.1002/9783527838707.ch4>.
33. Ai, H., S. Jian, Y. Yao, et al., “Aligned Porous Chitosan/Ti₃C₂ MXenes Monolith as a High-Performance Fixed-Bed Separation Medium toward Globular Albumin Separation,” *Chemical Engineering Journal* 461 (2023): 141913. <https://doi.org/10.1016/j.cej.2023.141913>.

34. Ding, L., Y. Wei, Y. Wang, et al., “MXene-Based Membranes: Synthesis, Properties, and Emerging Applications,” *Chemistry of Materials* 32 (2020): 1703–1747.
<https://doi.org/10.1021/acs.chemmater.9b04408>.