

Effect of MAX Phase $\text{Mo}_2\text{TiAlC}_2$ on the PVDF Ultrafiltration Membrane Properties and Performance

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Abstract: Ultrafiltration membranes are widely used in wastewater filtration due to their efficiency relative to conventional water treatment technologies. To improve the antifouling property of the PVDF membrane, a composite ultrafiltration membrane was fabricated employing the *in-situ* embedment approach throughout the phase inversion process and utilizing a new 2D material, MAX phase Molybdenum Titanium Aluminium Carbide ($\text{Mo}_2\text{TiAlC}_2$). The membranes were described using Fourier transform infrared spectroscopy (FTIR), Scanning electron microscopy (SEM), and porosity measurements. Rejection tests were applied to study the produced membranes. Adding $\text{Mo}_2\text{TiAlC}_2$ increased the hydrophilicity of the composite membrane compared to the pristine membrane. Porosity and membrane pore size increased with the addition up to 0.6% wt. The most hydrophilic membrane (M3) recorded the highest protein rejection of 84.9%, which was much higher than that of the pristine membrane. These findings highlight the potential of $\text{Mo}_2\text{TiAlC}_2$ as a promising PVDF membrane additive.

Keywords: Hydrophilicity, MAX phase $\text{Mo}_2\text{TiAlC}_2$, ultrafiltration, PVDF, membrane.

1. INTRODUCTION

Ultrafiltration (UF) is a low-pressure membrane process widely applied in water treatment due to its high separation efficiency and compact design [1]. UF membranes, which are available in both polymeric and ceramic forms, effectively remove various pollutants and microorganisms from water [2]. The technology has found applications across diverse industries, including textiles, dairy, and pharmaceuticals [2]. Recent advancements have focused on the development of tight UF membranes with smaller pore sizes (300-5000 Da) to improve the rejection of trace organic compounds and reduce fouling [3]. However, challenges such as low water flux and high operating pressure still need to be addressed [3]. UF membranes have become increasingly important in drinking water production as a safety barrier against bacteria and viruses, and in wastewater treatment for solute separation and water reuse [4]. The growing demand for improved membranes is driven by the increasing prevalence of water pollution and the associated treatment challenges.

Polyvinylidene fluoride (PVDF) is one of the most widely used materials for ultrafiltration membranes owing to its excellent properties. PVDF exhibits high mechanical strength, good thermal and chemical stability, resistance to aging, and favorable film-forming ability [5, 6]. These characteristics make PVDF

membranes suitable for various water treatment applications. However, the hydrophobic nature of PVDF leads to membrane fouling issues, which is a significant limitation [7, 8]. To address this challenge, researchers have developed various modification strategies to enhance the hydrophilicity and antifouling performance of PVDF membranes. These include surface coating, blending with hydrophilic materials, and chemical modifications such as amination and grafting [7, 8]. These modifications aim to improve membrane wettability, reduce organic fouling, and enhance overall filtration efficiency while maintaining PVDF's desirable properties [6, 8].

Membrane modification techniques can be broadly categorised into surface modification, physical blending (or doping), and chemical grafting [9]. Surface modification techniques involve hydrophilization, heating, and coating [9]. Chemical grafting can be achieved through "rafting-to" or "grafting-from" techniques [9]. Physical blending involves incorporating additives into the membrane matrix during synthesis [10]. Nanomaterial incorporation is a widely used and effective method due to its simplicity, cost-effectiveness, and minimal structural disruption [9]. Various nanomaterials, such as metal nanoparticles, zeolites, and carbon nanotubes, have been successfully integrated into membranes [9]. Other modification techniques include plasma treatment, surfactant modification, and polymer blending [10]. These modifications aim to improve membrane hydrophilicity, reduce fouling, and enhance overall performance [11, 12]. Emerging materials like polydopamine offer promising opportunities for membrane surface modification [12].

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MAX phases are layered ternary carbides/nitrides with a general formula $M_{n+1}AX_n$, where M is a transition metal, A is an A-group element, and X is carbon or nitrogen [13]. Their structure consists of $M_{n+1}AX_n$ sheets interleaved with A layers, resulting in a combination of metallic and ceramic properties [14]. MXenes, which are two-dimensional derivatives of MAX phases, are obtained by selectively etching the A layer through chemical treatment [13]. $\text{Mo}_2\text{TiAlC}_2$ is a specific MAX phase compound that can be transformed into $\text{Mo}_2\text{TiAlC}_2$ MXene [15]. MXenes exhibit high hydrophilicity and electrical conductivity [13]. $\text{Mo}_2\text{TiAlC}_2$ demonstrates mechanical stability and metallic electrical conductivity, with Mo 4d states dominating at the Fermi level [16]. The derived $\text{Mo}_2\text{TiAlC}_2$ MXene shows improved catalytic activity for hydrogen evolution reaction over time, attributed to MoO_2 formation [15].

Recent studies have demonstrated the effectiveness of incorporating nanofillers like graphene oxide (GO) and MXene into PVDF membranes to enhance their performance. These modifications have led to increased water permeability, with flux improvements ranging from 48% to 240% compared to pristine PVDF membranes [17-19]. The addition of nanofillers also significantly improved membrane hydrophilicity, as evidenced by reduced water contact angles [17, 20]. Furthermore, the modified membranes exhibited enhanced antifouling properties and dye rejection capabilities. For instance, GO-ZnO/PVDF membranes showed a substantial reduction in irreversible fouling ratio for bovine serum albumin [18], while MXene-modified GO/PVDF membranes demonstrated 100% removal of crystal violet dye [20]. These improvements in membrane performance were attributed to the synergistic effects of the nanofillers, including their high specific surface area and oxygen-containing functional groups [17].

2. MATERIALS AND METHODS

2.1. Materials

Polyvinylidene fluoride (PVDF) ($M_w = \sim 534,000$, Sigma-Aldrich, product no. 427152), polyvinylpyr-

rolidone (PVP) ($M_w = \sim 40,000$, Sigma-Aldrich, product no. PVP40), N-methyl-2-pyrrolidone (NMP) (Carl Roth, product no. 4306.1, $\geq 99\%$), and $\text{Mo}_2\text{TiAlC}_2$ MAX Phase powder (200mesh, 99%) was obtained by BEIJING 33ENE TECHNOLOGY CO., LTD, China. All chemicals were of analytical grade and used without further purification.

2.2. Membrane Preparation

The first preparation was made at room temperature, with stirring for 2 hours to homogeneously distribute the $\text{Mo}_2\text{TiAlC}_2$ MAX Phase nanoparticles in the solvent. After that, the stirring rates were gradually increased from 800 rpm to about 1600 rpm to enable proper dispersion of the nanoparticles. After dispersion, the vessel containing the mixture was then put into a water bath at about 60°C . This controlled heating ensured the gradual dissolution of the PVDF pellets and PVP powder into the stirring NMP solution. While the polymers were dissolving, the viscosity of the solution gradually increased, naturally reducing the stirring speed. Careful handling resulted in a homogeneous and well-dispersed resultant mixture ready for membrane casting. This was then poured on the borosilicate plate and compressed with another plate to yield the membrane. The thickness of the membrane was controlled by the Scotch tape method, where a number of superimposed layers of Scotch tape provide a particular thickness. This was to ensure that the compressed solution was completely soaked in distilled water for two days for the complete solidification of the membrane and to ensure that there were no residual solvents and impurities left behind. The Scotch tape method was employed as an alternative phase inversion technique due to the unavailability of a Doctor Blade, while still ensuring precise control over the membrane thickness. Further, post-treatment was carried out by dipping the membrane for two days in isopropanol and then for two days in glycerol to enhance its stability and keep it from becoming brittle. The treated membrane was air-dried and stored for filtration applications.

Table 1: Composition of Samples with Varying MAX Phase $\text{Mo}_2\text{TiAlC}_2$ Content

Sample Code	PVDF (%wt)	PVP (%wt)	NMP (%wt)	MAX Phase $\text{Mo}_2\text{TiAlC}_2$ (%wt)
M1	20	2	78.0	0.0
M2	20	2	77.6	0.4
M3	20	2	77.4	0.6
M4	20	2	77.2	0.8

*Composition percentages are by weight.

The MAX Phase concentration was optimized within the range of 0.1 wt% to 0.9 wt%, based on findings from previous research [21, 22].

2.3. Membrane Characterization

The morphology and the cross-section of the membranes were studied using a ZEISS EVO 15 Scanning Electron Microscope (SEM). Functional groups and chemical interactions of the membranes before and after incorporating $\text{Mo}_2\text{TiAlC}_2$ MAX Phase and filtration were analyzed by Fourier transform infrared (FTIR) spectroscopy. Fourier transform infrared (FTIR) spectroscopy PerkinElmer Spectrum Two Fourier-transform infrared spectrometer (FTIR) was used to confirm the successful incorporation of $\text{Mo}_2\text{TiAlC}_2$ MAX Phase and to analyze any chemical interactions between the membrane components. The membrane porosity was measured using the equation 1 [21],

$$\varepsilon\% = \frac{W_w - W_d}{A \times L \times \rho} \quad (1)$$

The W_w was determined by immersing a piece of membrane in distilled water for 24 h, removing the

excess water using blotting paper, and measuring its weight [21]. The W_d was measured by oven drying the same membrane samples for 12 h and measuring their weights [21].

where:

W_d = the membrane's dry weight (g)

W_w = the membrane's wet weight (g)

A = membrane area (cm^2)

ρ = water density = 1 g/cm^3

L = thickness of the membrane (cm)

3. RESULTS AND DISCUSSION

3.1. Characterization of PVDF/ MAX Phase $\text{Mo}_2\text{TiAlC}_2$ Membrane

3.1.1. Membrane Morphology and Porosity

The surface morphologies of the membranes (M1, M2, M3, and M4) were analyzed using SEM and the average pore distances were measured with ImageJ software. The Figure 1a, 1b, 1c, and 1d exhibit the

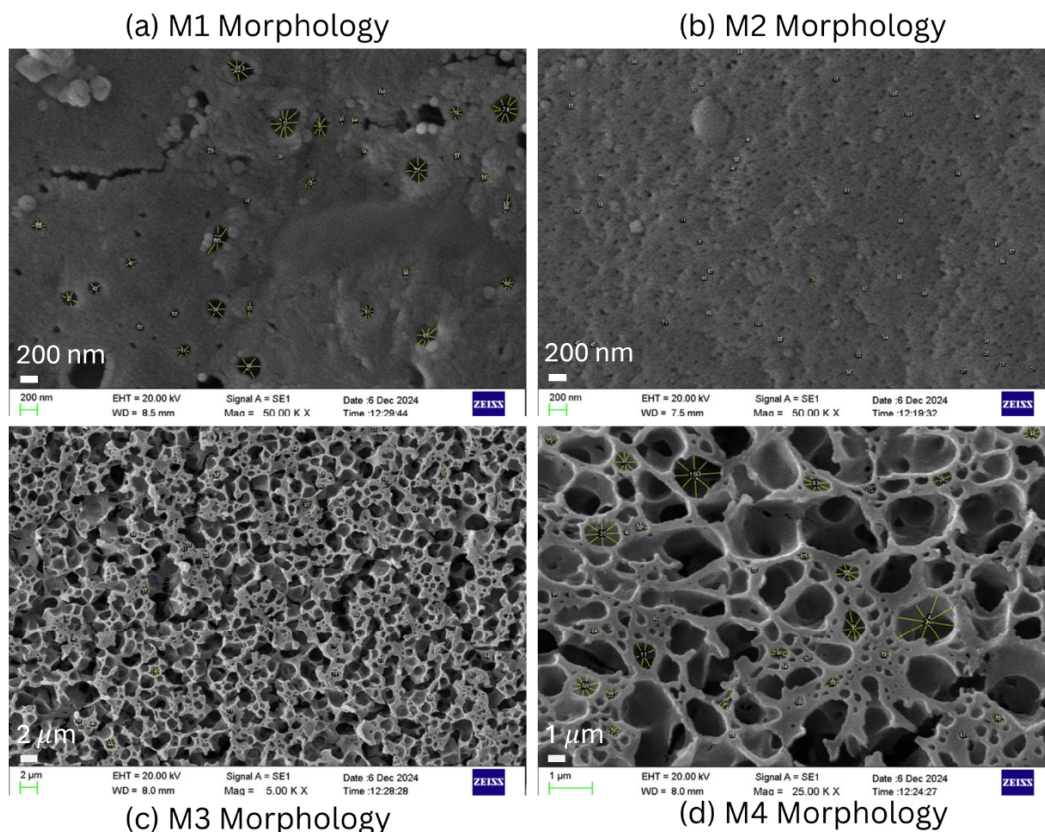


Figure 1: Surface morphology of the fabricated PVDF and $\text{Mo}_2\text{TiAlC}_2$ /PVDF composite membranes observed under SEM, showing the effect of MAX phase incorporation on membrane structure.

surface morphology. The mean pore size values of M2, M3, and M4 are, respectively, 30nm, 650nm, and 1144nm.

The M1 membrane, which was unmodified, had unevenly distributed pores and cracks that tended to appear. In some places, aggregations were also seen. With the addition of $\text{Mo}_2\text{TiAlC}_2$ MAX Phase, we can clearly see a pore formation that is evenly distributed than M1. The pore sizes are small, which is in the nano range. The M3 membrane has an increment in the pore size than the M2 membrane, and M4 has the largest pore size. All 4 membranes' thickness is kept in the 30 μm to 50 μm range. These results indicate that pore formation and pore size increase with the incorporation of $\text{Mo}_2\text{TiAlC}_2$ MAX Phase, consistent with observations reported for other MAX Phase-based ultrafiltration membranes [21, 22].

The porosity has an optimum condition even though the pore size has increased, as shown in Figure 2. The highest porosity can be seen in the M3 membrane. While the lowest can be seen in the M2 membrane. The porosity and pore size were not assessed in the M1 membrane due to the aggregations and cracks in the membrane. From Figure 2 it is clear that with the increment of $\text{Mo}_2\text{TiAlC}_2$ MAX Phase, the Pore Size increases, but not the porosity. By this result, we can clearly identify that, even though the size of a single pore increases due to the incorporation of the MAX Phase number of pores would not increase.

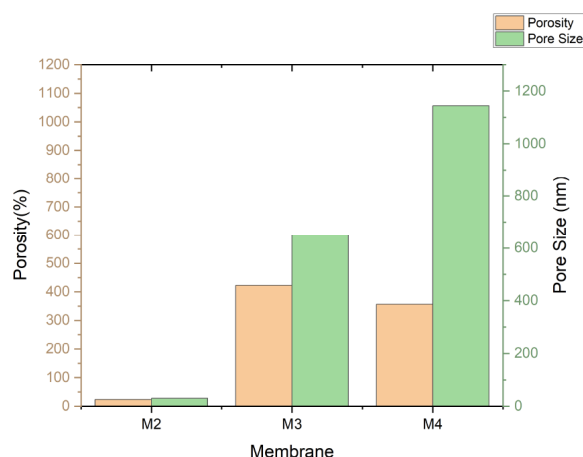


Figure 2: Variation of membrane porosity (%) and average pore size (nm) as a function of $\text{Mo}_2\text{TiAlC}_2$ loading, illustrating the influence of MAX phase content on membrane morphology.

3.1.2. Membrane Chemical Composition and Molecular Structure

The chemical composition and molecular structure analysis were conducted using a Fourier Transform

Infrared Spectroscopy. The Figure 3 depicts the FTIR spectra before filtration of Bovine Serum Albumin. The peak observed was at 1402 cm^{-1} , which corresponds to the stretching vibration of C-H [21]. The peak appeared at 1178 cm^{-1} corresponds to the vibrational modes of C-F bonds [21]. These peaks are a sign of the presence of C-H and C-F functional groups, as would be expected in the PVDF polymer [21]. These two peaks showed no noticeable change with the incorporation of the MAX Phase, confirming that the structural integrity of the PVDF matrix remained unaffected.

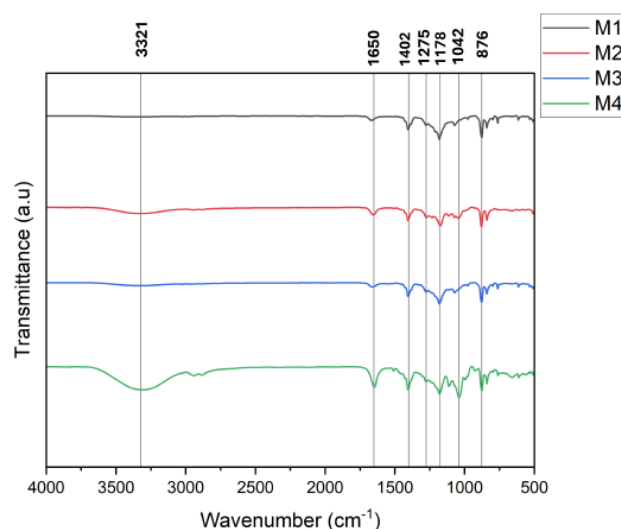


Figure 3: FTIR spectrum of the membranes before filtration, indicating the characteristic functional groups of PVDF and the presence of $\text{Mo}_2\text{TiAlC}_2$ -related peaks.

The peak at 1650 cm^{-1} depicts the -C=O bonds, and the peak at 1275 cm^{-1} depicts the C-N bonds [17, 23]. The peaks are formed due to the successful deposition of PVP, and these bands are characteristic of the acylamino functional groups present in the PVP structure [23]. The hydrophilicity of the membranes was enhanced by the addition of PVP. This is because the hydrophilicity was derived from the interactions of polar attractive forces and hydrogen bonds between acylamino and water molecules [23]. The peaks at 876 and 1042 cm^{-1} correspond to C-C and C-O stretching vibrational modes [24]. When the membranes before and after filtration were compared, a reduction in these two peaks was observed, indicating that the hydrophilic characteristics imparted by PVP were diminished after filtration.

The peak at 3321 cm^{-1} , corresponds to N-H stretching vibration, indicating the presence of amine or amide functional groups prior to filtration [25]. This peak is formed before filtration and then vanishes after

filtration. This is due to Catalytic N-H Bond Activation and Breaking. Transition metal complexes, such as those present in the $\text{Mo}_2\text{TiAlC}_2$ MAX phase, can weaken and activate N-H bonds by coordinating to them, making the bonds more reactive and easier to break, which can result in new bond formation and the loss of the original N-H bond [25].

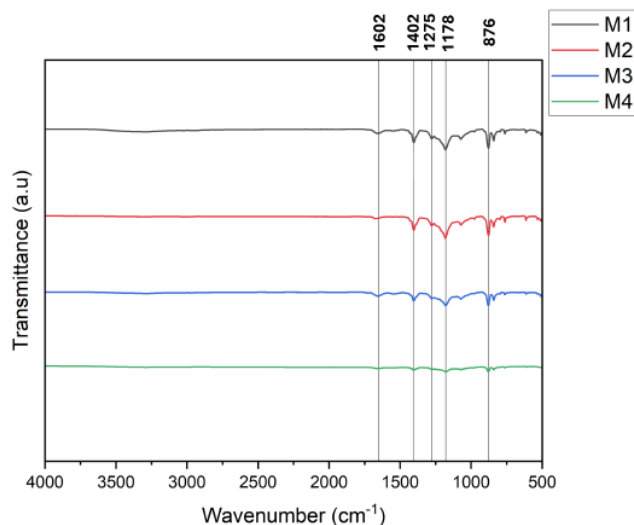


Figure 4: FTIR spectrum of the membranes after filtration, highlighting potential changes in functional groups and surface chemistry following protein filtration.

3.2. Membrane Performance

3.2.1. Bovine Serum Albumin (BSA) Rejection

A homemade dead-end filtration apparatus was used to determine the BSA rejection ratio. A 1000mg/L BSA concentration solution was used as the feed. The UV-Visible Spectroscopy was performed to find the

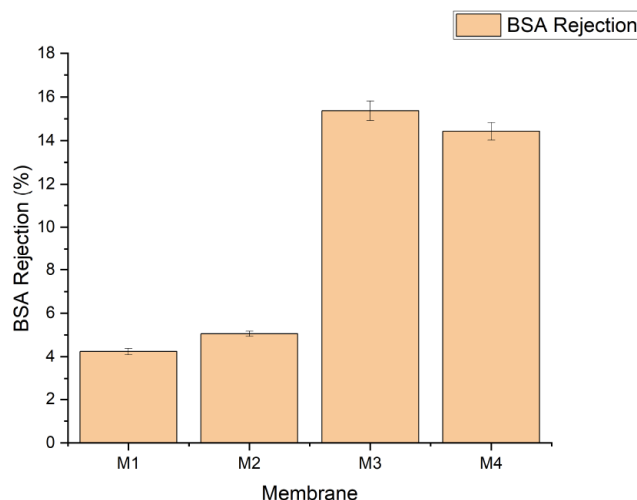


Figure 5: BSA rejection performance of neat PVDF and $\text{Mo}_2\text{TiAlC}_2$ /PVDF composite membranes, demonstrating the effect of MAX phase addition on separation efficiency.

concentration of the solution before and after filtration. The M1 and M2 membranes have the lowest rejection, while M3 has the highest rejection. The reason for the better rejection of M3 and M4 can be seen from Figure 4. After filtration, the M1 and M2 membrane peaks decreased slightly, which resulted in lower BSA rejection. The peaks in M3 and M4 have almost vanished and have a drastic BSA rejection. However, the M3 membrane exhibited the highest protein rejection of 84.9%, which is slightly lower than the BSA rejection reported for the Ti_3AlC_2 MAX phase/PVDF membrane (90.6%) [21].

4. CONCLUSION

This study utilised the composite MAX phase $\text{Mo}_2\text{TiAlC}_2$ to synthesise PVDF membranes for ultrafiltration applications. Different amounts of $\text{Mo}_2\text{TiAlC}_2$ were incorporated to determine the optimum nanoparticle-to-polymer ratio. Various analytical techniques were employed to examine and monitor the structural and performance changes induced by the additive. The performance of the nanocomposite membranes was evaluated through BSA rejection tests. The incorporation of $\text{Mo}_2\text{TiAlC}_2$ increased the mean pore diameters of the membranes to approximately 30 nm, 650 nm, and 1144 nm, respectively. However, these effects were significant only up to a concentration of 0.6 wt%. Among all samples, the M3 membrane exhibited the highest BSA rejection of 84.9%. The findings of this study demonstrate the potential of $\text{Mo}_2\text{TiAlC}_2$ as an effective additive for minimising PVDF membrane fouling, warranting further investigation into its long-term performance and applicability in complex feed systems. Future work will focus on conducting extended filtration experiments to assess membrane durability and antifouling behaviour over time, as well as exploring the performance of the $\text{Mo}_2\text{TiAlC}_2$ /PVDF mixed matrix membranes in treating actual wastewater samples to validate their practical applicability under realistic operating conditions.

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