



Iranian Research Organization
for Science and Technology
(IROST)

Advances
Environmental
Technology



Journal home page: <https://aet.irost.ir>

High-performance polysulfone/NH₂-MIL-125 membranes for the rejection of toxic metals in aqueous solutions

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ARTICLE INFO

Document Type:
Research Paper

Article history:
Received 02 October 2025
Received in revised form
10 February 2026
Accepted 20 February 2026

Keywords:
Metal-organic framework
Membrane
Heavy metal removal

ABSTRACT

Water contamination is a significant environmental issue, and it is crucial to develop innovative technologies to address this problem. One such technology is the use of nanomaterials in polymeric membranes, which can help to purify water by eliminating pollutants and heavy metals. These membranes possess exceptional properties, including a large surface area, adjustable pore dimensions, and permeability selectivity, which make them effective in removing various contaminants from water. Nanoscale materials, like metal nanoparticles, nanofibers, graphene, and graphene oxide, and metal organic framework (MOF), are integrated into the membrane, which enhances its mechanical strength, separation efficacy, and adsorption capabilities. In the current investigation, we have successfully synthesized the metal-organic framework NH₂-MIL-125 and conducted preliminary research on its properties for heavy metal rejection (Pb²⁺ and Cd²⁺) after incorporation into a polysulfone membrane. Field Emission Scanning Electron Microscopy (FESEM), X-ray Diffraction (XRD), Brunauer-Emmett-Teller (BET) analysis, an Electrokinetic Analyzer, and Fourier Transform Infrared Spectroscopy (FTIR) were used to study the membranes. Additionally, the membrane's water affinity, flow rate, and resistance to fouling were studied. The M-3 membrane with 3.0 % MOF incorporation showed a 99.10 % rejection for cadmium, and the M-3 membrane rejected 75.02% for lead at a feed concentration of 500 ppm.

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DOI: 10.22104/aet.2026.7601.2133

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1. Introduction

As global freshwater demand has risen to its highest level in the past decade, and the subsequent decrease in freshwater levels both above and below the Earth's crust has diverted mankind's attention to invent new technologies for water purification. Among the different techniques explored in the field of water purification, membrane technology is found to be one of the most promising and successful techniques. Nanofiltration (NF), ultrafiltration (UF), Microfiltration (MF), and Reverse Osmosis (RO) are extensively applied techniques across various countries worldwide. There has been a great improvement in the quality of life with the help of polymer science, membrane technology, and development in engineering fields [1, 2].

Elements with atomic masses between 63.5 and 200.6 and a specific gravity greater than 5.0 are classified as heavy metals. Though biological entities require a trace amount of heavy metal for the body's functions, an excess amount of it leads to fatality. The main sources of non-essential heavy metals are various industrial sectors, and wastewater from these industries is often directly discharged into freshwater sources, leading to contamination of drinking water [3]. During the initial stage, humans can tolerate small amounts of heavy metals entering their bodies; however, severe repercussions will be observed due to long-term exposure or injection. Hence, the World Health Organization (WHO) has established maximum contamination levels (MCLs) for the percentage of heavy metals in water. Above 10 ppb, 3 ppb, 10 ppb, and 100 ppb for Lead, Cadmium, Arsenic, and Chromium in drinking water are considered to be highly unsafe for human consumption [4]. However, some of them require the body's nutrition, but the ionic form of these metals is toxic due to their non-decomposable and accession nature inside the body. Many health issues related to body organs, biological cycles, and physiology have been reported due to these heavy metals, which subsequently lead to fatal diseases like cancer, mutagenicity, or genotoxicity [5]. Numerous technologies have been developed for extracting heavy metals from wastewater. The universally developed techniques for elimination of

contaminants from water, like adsorption[6-8], oxidation using various oxidants[9-11], membrane technology [12-14], ozonation[15-17], ion exchange[18, 19], chemical precipitation[20; 21], electrodialysis [22, 23], photo-catalytic degradation[24, 25], electrocoagulation[26-28], other techniques involving chemical and biological procedures[29-33] have been widely used across various sectors. Membrane technology has been extensively adopted in various industries due to its high-quality results, reduced chemical usage, low operating costs, decreased energy consumption, smaller footprint, and simplified scaling. The high operational costs, high waste generation, high energy consumption, and lower efficiency have led to a diversification of attention towards more efficient membrane technology [34]. Conventional membranes currently hold a predominant position in the water purification market; however, their selectivity, permeability, flux, stability, and high manufacturing costs make them less attractive for commercialization [35, 36]. These membranes are made up of various polymeric materials such as polyvinylidene fluoride (PVDF), polyether sulfone (PES), polypropylene (PP), polyvinyl chloride (PVC), polyacrylonitrile (PAN), polysulfone (PSF), polyamide, polyethylene (PE), and polyvinyl alcohol (PVA), based on the intended use[37]. The assimilation of nanoparticles into the membrane significantly enhances the membrane's ability to remove heavy metals[38].

Recently, several 2D and 3D nanomaterials have been incorporated into water purification techniques. This significant development in the field of nanomaterials is attributed to their exceptional physical and chemical properties, which significantly enhance the separation capabilities of polymeric membranes. The unique characteristics of nanomaterials present in polymeric membranes, such as their large surface area, adjustable pore sizes, and ability to selectively allow certain particles to pass through, have made significant advancements in removing various types of contaminants from water. The inclusion of nanoscale materials such as metallic nanocrystals, nanoscale fibers, Carbon nanostructures, and oxidized graphene has greatly improved membrane capabilities by enhancing mechanical strength, improving separation

efficiency, and increasing adsorption capabilities [39]. Different nanoparticles, such as metal-organic frameworks (MOFs) [40], covalent organic frameworks (COFs) [41], graphene oxide [42], zeolites [43], transition metal dichalcogenides [44] and MXenes [45] have been used in water purification.

Researchers in the field of water purification technology are increasingly interested in metal-organic framework-induced mixed matrix membranes. This is a result of the extensive surface area, multiple potential applications, and different pore sizes and functionalities of the nanofillers (MOFs). The organic ligand and the inorganic metal ion or cluster, which are the two components of MOFs, have a synergistic effect that provides exceptional properties, such as variable pore dimension, inner surface spaces, and massive porosity [46, 47]. The MOF-induced mixed matrix membranes have been synthesized and utilized in various applications, including gas separation, water treatment, and fluid separation. They have been used, especially in removing dense metals such as lead and arsenic, from effluent [37]. Titanium-based MOFs have been found to be highly beneficial in water purification technology. They have been widely used in the removal of heavy metals and dyes from industrial and other wastewater sources [48]. It has been reported that the modified NH₂-MIL-125, a titanium-centric metal-organic framework featuring an amine organic functional group, exhibits adsorption properties of 102.8 and 66.9 mg/g for lead and cadmium, respectively [49]. The MIL-125 MOFs were also found to enhance water flux and remove pesticides from wastewater resources [50, 51].

In our earlier work, polysulfone mixed matrix membranes containing MIL-125(Ti) were fabricated and evaluated for the removal of organic dyes (Reactive Black 5 and Reactive Orange 16) and heavy metals (lead and cadmium). The membranes exhibited rejection efficiencies of 90% for RB-5, 47% for RO-16, 89.33% for Pb(II), and 68.81% for Cd(II), confirming the potential of MIL-125(Ti) as a suitable additive for enhancing performance. The monographs describing the membrane morphology and performance are included in the previously published work [52]. Building on these findings, the present study

investigates the use of the functionalized analogue, NH₂-MIL-125(Ti), in PSF-based mixed matrix membranes, explicitly targeting the removal of heavy metals. The incorporation of amino-functionalized MIL-125(Ti) significantly improved the rejection efficiency, achieving 99.1% for Pb(II) and 75.02% for Cd(II), thereby demonstrating the superior performance of the modified MOF compared to its parent structure.

There are reports of the usage of NH₂-MIL-125 for light-catalyzed decomposition of dyes [53], oil and water emulsion separation [54], gas separation [55], desalination [56], Medical [57], and heavy metal elimination [58]. In this research, the MOF has been synthesized using a solvothermal method [59]. The polymeric material Polysulfone (PSF) is a commercially available glassy material used for the construction of UF [60] and NF [61] membranes. These polymeric materials are highly resistant to thermal and chemical conditions, exhibiting excellent mechanical strength. They are hydrophobic, which causes an increase in membrane fouling [62]. Therefore, various routes have been employed to enhance the flux and removal efficiency, while also decreasing the fouling nature. This study focuses on fabricating NH₂-MIL-125/ PSF composite membrane and its efficiency in removing Pb²⁺ (lead) and Cd²⁺ (cadmium) heavy metals.

2. Materials and methods

2.1. Reagents

All reagents and compounds used were of laboratory-grade quality and were applied directly without further refinement. A polysulfone polymer possessing an approximate molar mass of 35 kDa was procured from Solvay Industry. 1-methyl-2-pyrrolidinone and poly(1-vinyl-2-pyrrolidone) were purchased from Spectrochem, India. Fraction V bovine plasma protein was obtained through HiMedia industry. Titanium (IV) isopropoxide [Ti(OCH(CH₃)₂)₄, 95% purity], N, N-methanoyl dimethylamine, and benzene-1,4-dicarboxamide-2-amine were received from Merck India. Loba Chemie, India, delivered methyl alcohol, whereas cadmium nitrate tetrahydrate (98%) and lead nitrate (99.0%) were supplied by Sigma-Aldrich.

2.2. Synthesis of metal-organic framework $\text{NH}_2\text{-MIL-125}(\text{Ti})$

The synthesis of $\text{NH}_2\text{-MIL-125}$ was conducted following the earlier reported technique with minor modifications [63]. In brief, 0.006 M 2-Amino Terephthalic acid was dissolved in N, N-dimethylformamide (DMF) and stirred for 10 minutes to obtain a clear solution. Then, about 0.003 M titanium isopropoxide was added slowly and stirred to create a homogeneous mixture. Then, approximately 25 mL of methanol was added to the reaction mass and stirred for 30 minutes to form a homogeneous mixture. Then, the reaction

mixture is transferred to a 65 mL Teflon-coated stainless-steel autoclave and subjected to heating at 150 °C for a duration of 16 h. Once the reaction was complete, it was brought to room temperature and subsequently rinsed twice with 100 mL of DMF, followed by 100 mL of methanol. The resulting yellow-colored product was dried at 60 °C until it was free from solvents. The obtained material is used as a nano-additive with the PSF polymer to prepare the mixed matrix membranes. Figure 1 represents the synthetic route for the preparation of $\text{NH}_2\text{-MIL-125}$ [64], and Figure 2 depicts the structural representation of $\text{NH}_2\text{-MIL-125}$ [65].

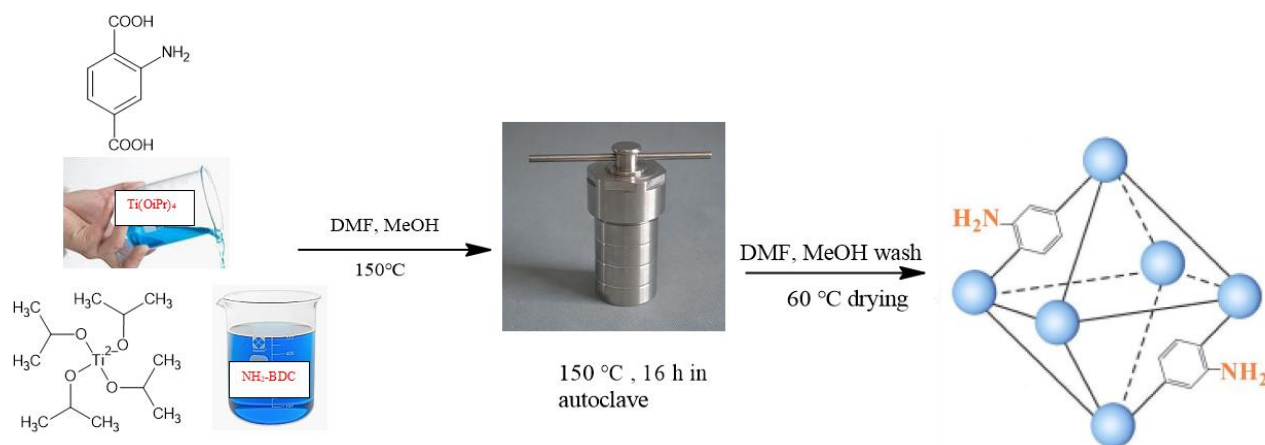


Fig. 1. Schematic representation of the route of synthesis of $\text{NH}_2\text{-MIL-125}$ MOF

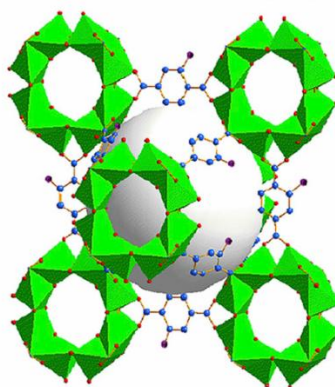


Fig. 2. Structure of $\text{NH}_2\text{-MIL-125}$

2.3. Fabrication of $\text{NH}_2\text{-MIL-125/PSF}$ composite films.

The $\text{NH}_2\text{-MIL-125/PSF}$ nanocomposite thin film was fabricated by a nonsolvent-induced phase separation method [66]. To initiate the experiment, the PSF material was desiccated at 60 °C under reduced pressure for one day to ensure

that no moisture content remained. Then, fixed quantities of $\text{NH}_2\text{-MIL-125}$ (0 wt%, 0.5 wt%, 1 wt%, and 3 wt%) with respect to the weight of polymer were added to N-Methyl-2-pyrrolidone (NMP) solvent in a reagent bottle. The mixture was sonicated for 0.5 h at ambient temperature using a

40 kHz, 60W Spectra lab sonicator to get a clear dispersion.

Then, the PSF polymer and 1% Polyvinylpyrrolidone (PVP) were incorporated into the MOF solution in NMP and agitated at 60°C for 1 day to obtain a transparent and consistent casting solution.

The dope solution was degassed to eliminate gas bubbles, and the polymer solution was then cast onto a transparent disc using a K-202 precision coating unit. The casted transparent panel was kept still for 10 s before being transferred to a coagulation chamber filled with water that had its minerals removed. The resulting membranes were left undisturbed in deionized water (DM) for 24 h. The specifics of the thin film formulation are provided in Table 1.

2.4. Description of NH₂ - MIL-125 and the blended nanocomposite films of NH₂ - MIL-125/PSF. Surface structures of NH₂ - MIL-125 and NH₂ -MIL-125/PSF composite membranes.

The morphological structure of the prepared nanocomposite and the mixed matrix membranes was analyzed using a Field Emission Scanning Electron Microscope (FESEM) instrument. The chemical composition of the sample was determined using energy-dispersive X-ray spectroscopy. The framework crystals were spread out on a graphite adhesive strip, and a fine coating of gold was applied to the surface. FESEM images were then taken for analysis. The thin film substance was dipped into cryogenic fluid for around one min, after which it was fractured using a knife-edge. Next, the material was mounted on a

mounting platform, coated with gold colloids via a sputtering apparatus, and then studied for modifications observed across sections with the aid of an FESEM [67]. The size, structure, orientation, and extent of crystallinity, as well as information on unit cell dimensions, of the nanocomposite material were determined using X-ray Powder Diffraction (XRD) [62].

The occurrence of desired chemical groups in the nanocomposite and the proper blending of MOF into the polymeric membranes have been analyzed using FTIR and Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy (ATR-FTIR) techniques. About 0.02g of NH₂-MIL-125 was finely milled with anhydrous KBr particles to prepare the sample. The FTIR spectra for NH₂-MIL-125 and the ATR-FTIR spectra of the blended membranes were also analyzed [68, 69].

The stability with respect to temperature has been investigated using a thermogravimetric analyzer. The cumulative pore space and mean pore radius for NH₂-MIL-125 have been calculated as per the reported methods. The interfacial extent per mass for a nanoscale particle plus hybrid polymer barriers was computed [70] and the pore volume (P_v) was also calculated through reported methods [71].

A contact angle analyzer was used to study the surface hydrophilicity of the prepared composite films. The hydrophilicity of the surface was measured using the KYOWA (Japan) surface wetting measurement instrument [72].

Table 1. Dope solution composition used in the preparation of mixed matrix membranes.

Membrane	PSF (g)	NMP (g)	PVP (g)	MIL-125 (Ti) wt%	MIL-125 (Ti) (g)
M-0	1.6	8.2	0.016	0	0
M-1	1.6	8.192	0.016	0.5	0.008
M-2	1.6	8.184	0.016	1	0.016
M-3	1.6	8.152	0.016	3	0.048

2.5. Investigation of water absorption and the pore structure of hybrid membranes.

The investigation of the membranes' ability to absorb water and their porosity levels was conducted following the established procedure [73].

A membrane with an area of 1 cm² was soaked in demineralized water for a duration of 24 h. Once

the period was over, the membrane was removed from the liquid, and its moisture-laden weight was recorded. The wet film specimen was then placed in a vacuum oven at 60 °C for 24 h, after which its dry weight was noted. Based on this data, the percentage of water absorption was determined using the formula Eq. 1.

$$\% \text{ Water uptake} = \left(\frac{W_w - W_d}{W_w} \right) \times 100 \quad (1)$$

In the formula, the weight of the membrane in its dry state is considered W_d and the weight of the membrane in its wet state is regarded as W_w .

The determination of porosity has been carried out following Equation 2

$$\% \text{ Porosity calculation} = \left(\frac{C_w - C_d}{A \times \rho_w \times \delta} \right) \times 100 \quad (2)$$

where C_d refers to the dry mass of the prepared membrane, and C_w signifies the mass of the membrane when it is hydrated, ρ_w is the mass per unit volume of water (0.998 g cm^{-3}), A is the membrane area in wet condition (cm^2), and δ is the thickness of the membrane in wet condition.

2.6. Pure water permeability study of the fabricated membrane

Water filtration experiments were conducted in the laboratory to determine the pure water flux (PWF) of composite membranes made from pristine and MOF blends.

Circular membranes with a measuring area of 5 cm^2 were cut and placed in a sample holder for the compaction process at a pressure of 0.3 MPa for 30 minutes. Subsequently, the pressure was decreased to 0.2 MPa , during which the membranes were assessed for an additional 60 minutes. The inflow used was process water, and the flow rate of clean water was measured following Equation 3, as mentioned in prior research. [73].

$$L_w = \frac{V}{M_t} \quad (3)$$

where, L_w represents the flow rate of pure water ($\text{L/m}^2/\text{h}$), V denotes the overall amount of liquid gathered, M refers to the effective membrane surface area and t indicates the duration of the experiment.

2.7. Fouling resistivity study of the fabricated thin film membranes

The study investigated the antifouling properties of neat and hybrid thin films as per the previously reported method [74]. A 1000 ppm Bovine Serum Albumin (BSA) solution was used to conduct flux studies at a pressure of 0.2 MPa . Firstly, the permeability of pure water (K_{w1}) in $\text{L/m}^2/\text{h}$ was measured for 60 min. Then, the BSA permeability studies were conducted and reported as K_p in $\text{L/m}^2/\text{h}$. Subsequently, the membrane was rinsed extensively under a continuous flow of tap water

for 20 minutes. Finally, pure water permeability (K_{w2}) in $\text{L/m}^2/\text{h}$ was measured for an additional 60 min. The membrane's ability to resist fouling was determined using Equation 5.

$$\text{FRR}\% = \left(\frac{K_{w2}}{K_{w1}} \right) \times 100 \quad (5)$$

The greater the Flux Recovery Ratio (FRR%), the more effective the material is against fouling. Likewise, the measurements for reversible fouling (R_r), irreparable fouling (R_{ir}), and overall fouling ratio (R_t) have been measured using Eqs. (6)-(8)

$$R_r (\%) = \left(\frac{K_{w2} - K_p}{K_{w1}} \right) \times 100 \quad (6)$$

$$R_{ir} (\%) = \left(\frac{K_{w1} - K_{w2}}{K_{w1}} \right) \times 100 \quad (7)$$

$$R_t (\%) = \left(\frac{K_{w1} - K_p}{K_{w1}} \right) \times 100 \quad (8)$$

2.8. Investigation of the removal of harmful heavy metal ions.

Removal analysis of harmful heavy metals was conducted using the dead-end filtration method. Solutions of lead(II) nitrate and cadmium(II) nitrate in water were prepared at a concentration of 500 ppm, complexed with 1 wt% Polyethyleneimine (PEI), and studied under a pressure drop across the membrane of 0.2 MPa for one hour. Although both lead and cadmium are divalent in nature, the difference in ionic size, speciation, and affinity towards PEI will lead to strong and stable PEI-Cd complexes compared to PEI-Pb complexes. The hydrodynamic radius is also larger for the former compared to the latter. The rate of rejection was assessed by analyzing the concentration of heavy metals in both the source fluid and the collected filtrate using Atomic Absorption Spectroscopy (AAS) equipment. The refusal rate was computed using Equation 9 [38].

$$\%R = \left(1 - \frac{N_p}{N_f} \right) \times 100 \quad (9)$$

where N_p denotes the level of the permeate solution, while N_f indicates the level of the feed solution

3. Results and discussion

3.1. Description of NH₂-MIL-125: High-Resolution Electron Beam Surface Imaging System (FESEM) analysis

The structural appearance of the manufactured nanoparticles has been studied using FESEM images, and their elemental composition has been identified with the help of Energy-Dispersive X-ray spectroscopy (EDX). The FESEM images show granular-shaped nanoparticles with smooth surfaces. The cylindrical "cake" like structures exhibit a length of 700-800nm with a thickness of 200 nm [52].

The nanoparticle's shape may vary depending on the concentration of the reactants and the solvents used during the synthesis [59]. The EDX spectrum showed (Figure 3) that the elemental composition is comprised of titanium, carbon, nitrogen, and oxygen, affirming the formation of NH₂-MIL-125.

3.2. Fourier Transform Infrared Spectroscopy (FTIR) analysis

The FTIR spectrum of NH₂-MIL-125 is shown in Figure 4, displaying the characteristic peaks. All of the spectra display distinctive vibrational bands in the 1400–1700 cm⁻¹ region, characteristic of the carboxylate group within the titanium-linked metal-organic framework (MOF) architecture. The compound being analyzed exhibits several absorption bands in its infrared spectrum. Specifically, two absorption lines at approximately 1600 and 1500 cm⁻¹ can be attributed to uneven oscillations of carbonyl stretching. Meanwhile, bands around 1440 and 1400 cm⁻¹ indicate even oscillations of carbonyl stretching. Additionally, the band observed at 1250 cm⁻¹ is attributed to the C–H symmetric stretching vibrations of the benzene ring. The region between 400 and 800 cm⁻¹ features the vibrations of Ti–O–Ti–O. Finally, the bands at 3500 cm⁻¹ and 3380 cm⁻¹ indicate the presence of the NH₂ group [59].

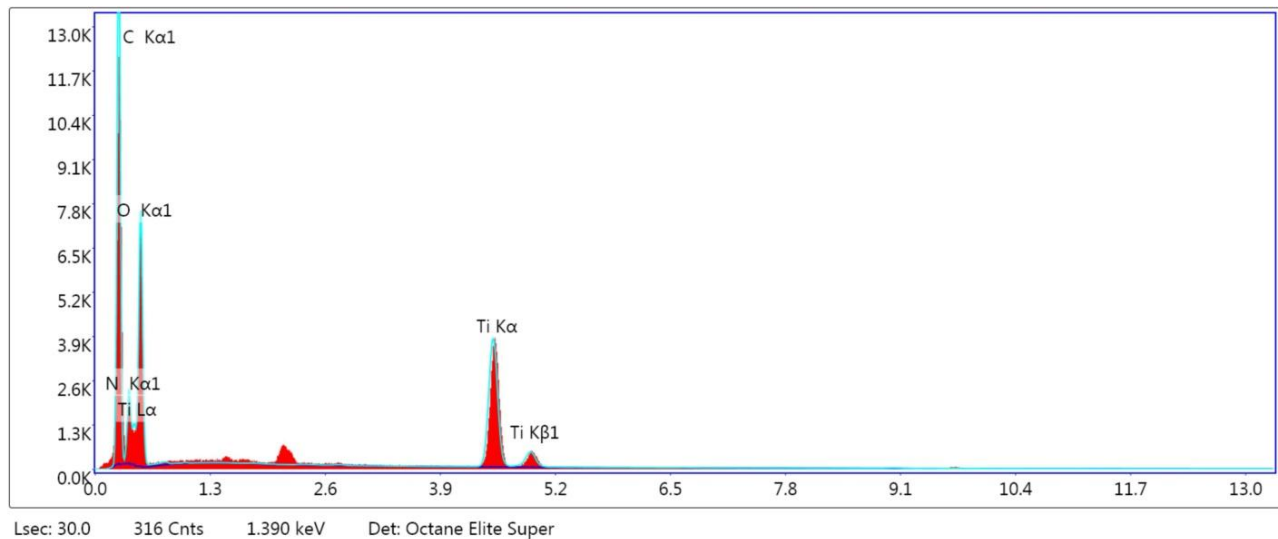


Fig. 3. Graphical representation of EDX analysis report of MIL-125(Ti)

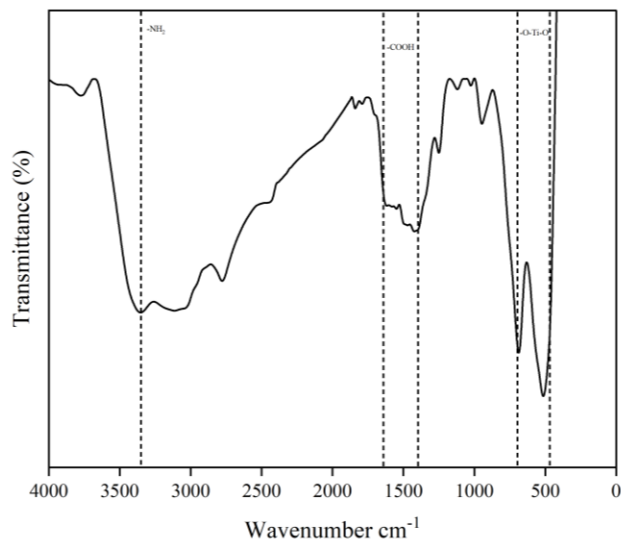


Fig. 4. IR spectrum of $\text{NH}_2\text{-MIL-125(Ti)}$ crystals synthesized

3.3. Thermal Weight Measurement (TWM) and Differential Thermal Examination (DTE)

The heat solidity of the produced MOF material (Ti) was assessed using TWM and DTE measurements. The initial reduction in weight noted between 80°C to 120°C corresponds to trapped solvents and water traces. Upon further heating, the degradation of amino terephthalic acid starts at around 320°C and goes up to 470°C , where the complete oxidation of organic linkers was observed [52]. Only the residue of metal oxide (TiO_2) will be observed above that temperature [59].

3.4. X-ray Powder Diffraction (XRD) Analysis

The crystal building of the synthesized nanomaterial was investigated using the Powder X-ray Diffraction (PXRD) technique.

From Figure 5, we can see all the crystalline patterns of the synthesized MOF. This structure consists of chains of corner-sharing TiO_6 octahedra with $\mu\text{-OH}$ groups, interconnected by $\text{NH}_2\text{-BDC}$ molecules to form a 3D porous network. The sharp peaks at 2θ of 6.71° , 9.58° , and 11.58° indicate that $\text{NH}_2\text{-MIL-125}$ has been formed as reported in the literature [68].

3.5. Gas Adsorption Surface Area Method (BET Theory) Assessment

The nitrogen adsorption-desorption isotherm of $\text{NH}_2\text{-MIL-125}$ metal-organic framework and the corresponding aperture dimension distribution are

studied. BET measurements indicated that the available surface area was $1042.04\text{ m}^2/\text{g}$.

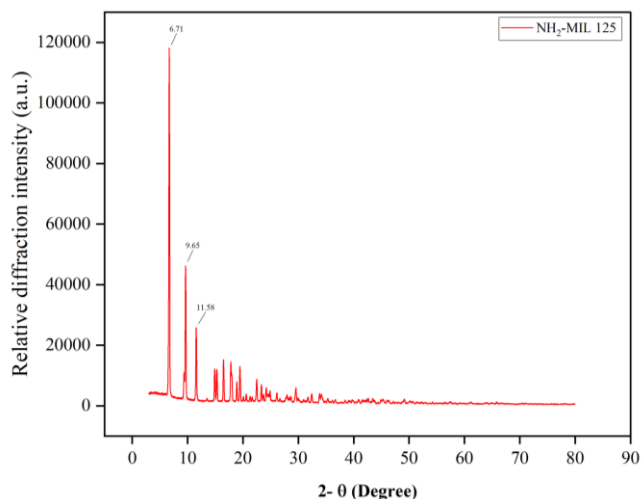


Fig. 5 The XRD pattern of $\text{NH}_2\text{-MIL-125}$

The adsorption pore diameter is found to be 1.4 nm , and for desorption, it is found to be 1.94046 nm . The pore volume is found to be $0.5\text{ cm}^3/\text{g}$. The graph indicates a type-III adsorption isotherm, which is flat due to the superposition of adsorption and desorption isotherms, mostly occurring in micropores [75].

The Barrett-Joyner-Halenda (BJH) pore size distribution of the synthesized MOF was similar to previously reported work [52]. The maximum point at 2.7 nm suggests a structure with tiny pores [76, 77].

3.6. Examinations of the structure of blended membranes Assessment using a Field Emission Scanning Electron Microscope (FESEM)

The surface topography of the original (M-0) and MOF-doped films was analyzed using the FESEM technique. The images show a top dense layer and a porous sublayer attributed to asymmetric membranes. The neat film (M-0) shows tiny digit extensions, and as we increase the percentage composition of nano components, the finger-like projections and the macrovoids increase from M-1 to M-3. The increase in spongy structure and macrovoids is owing to hydrophilic carboxylate groups and amino groups of MOF. The addition of a pore-generating agent, PVP, to the casting solution accelerates the phase inversion process, resulting in larger macrovoids. The EDX analysis (Figure 6 (a, b, c, d)) shows the occurrence of Nitrogen, Oxygen, Sulphur, Carbon, and Titanium on the membrane matrix of composite membranes, showing the successful incorporation of nanoparticles on the membrane surface [73, 78].

3.7. Fourier Transform Infrared Spectroscopy (FTIR) Analysis

The various functional moieties present in the PSF polymer and the incorporated MOF have been analyzed using the FTIR technique [53, 69]. Figure 7 shows the grouped spectrum of neat and mixed matrix thin films.

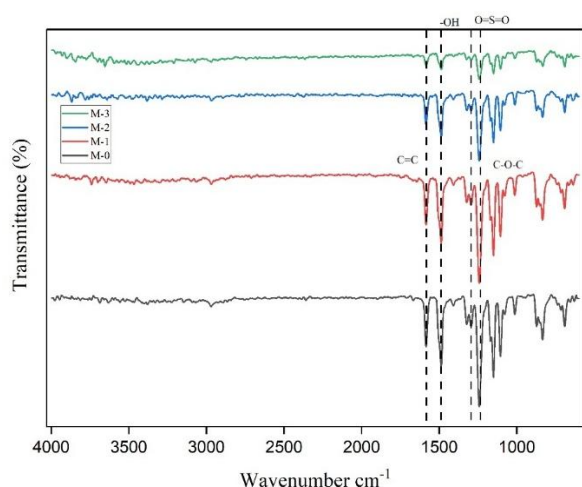


Fig. 7. The stacked FTIR spectra of both pristine and composite membranes

The peaks around 3400 cm^{-1} are attributed to the vibration of $-\text{OH}$ and $-\text{NH}$ groups. The maximum observed at 1590 cm^{-1} is linked to $\text{C}=\text{C}$ vibrations, while the prominent absorption peak at 1490 cm^{-1} is associated with $-\text{OH}$ deformation. The resonance bands detected at 1300 cm^{-1} and 1350 cm^{-1} are associated with the stretching frequencies of the $\text{O}=\text{S}=\text{O}$ group. The primary Ti-related peaks are observed in the region between 400 and 800 cm^{-1} . The Ti-O stretching vibration is observed at $650\text{--}800\text{ cm}^{-1}$, and the Ti-O-Ti bridging vibrations are observed at $650\text{--}800\text{ cm}^{-1}$. The Ti-O-Ti bridging stretching mode is also observed at 424 cm^{-1} [79-81].

3.8. Contact angle measurements

The surface absorbency, hydrophobicity, and hydrophilicity of the prepared membranes have been investigated using contact angle analysis. By employing the sessile drop technique, the contact angle is measured to determine the preferential wetting of a given membrane by water. The neat thin film M-0 exhibited a surface wettability measurement of 85.1° , while M-1 produced 75.5° , M-2 showed 69.9° , and M-3, with the least droplet interface angle of 65.5° . This indicates that the addition of nanoparticles has enhanced the water-attracting properties of the fabricated membranes [82].

3.9. Clean water flow analysis

Filtration studies have been conducted to assess the pure water permeability of the original and nanocomposite membranes. The M-3 film exhibited a maximum flux of $202.69\text{ L/m}^2/\text{h}$ after 60 mins, while the original membrane M-0 displayed a flux of $44.57\text{ L/m}^2/\text{h}$ after 60 mins.

3.10. Antifouling nature of membranes

During filtration studies, scientists consider fouling of prepared membranes a major issue. Several factors, including pore plugging, narrowing, and the formation of protein cake, cause this fouling. These factors are of major concern when preparing membranes for commercialization [83]. The M-3 film demonstrated the most significant resistance to membrane fouling.

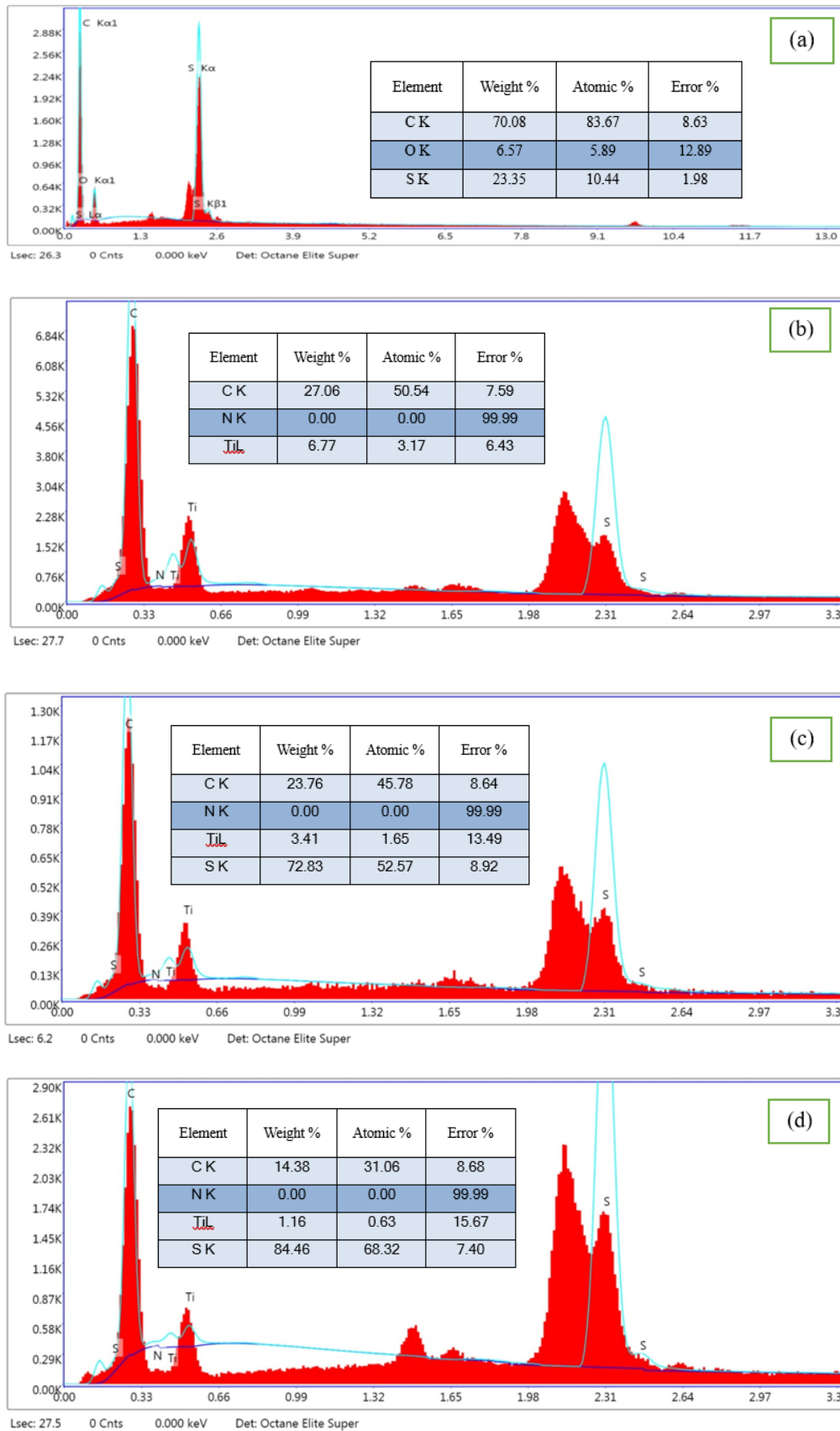


Fig. 6. The EDX outcomes for (a) M-0, (b) M-1, (c) M-2, and (d) M-3 are provided.

3.11. Investigation of moisture absorption and void space analysis

The effect of nanoparticles on the synthesized mixed matrix membranes is collectively displayed in Table 2.

The water uptake increased by 21.93% when transitioning from the pristine to the M-3 membrane, with the M-3 membrane exhibiting the maximum porosity.

The same trend has also been observed in contact angle measurements. The homogeneous distribution of MOF on the membrane matrix has led to the formation of more pores and voids, which in turn leads to an increase in flux, water uptake, and porosity values [84].

The MOF membrane has a relatively uniform and sturdy porosity, but the shape of its pores seems to be irregular and elongated.

These asymmetrical pore structures, which can resemble finger-like or spongelike projections, have the potential to boost mass transfer and, as a result, improve rejection efficiency [52]. The MOFs made of terephthalic acid and 2-amino terephthalic acid efficiently interact with membranes by acting as pore-forming agents [85].

3.12. Heavy metal rejection studies of membranes

In this study, pristine and MOF-amalgamated mixed matrix membranes were used to investigate the rejection capabilities at varying concentrations of MOFs.

The rejection studies have been depicted using a bar graph for lead and cadmium for different membranes.

Figure 8 displays the details of the rejection study. In this study, the M-3 composite film with 3.0% MOF incorporation exhibited a 99.10% rejection for cadmium and a 75.02% rejection for lead. In the

investigation of heavy metal filtration, agents such as polyethyleneimine (PEI) are employed to enhance the particle size of the metals, resulting in improved rejection rates.

The rejection percentage has increased by approximately 14% from the pristine membrane to the 3% MOF-induced membrane for cadmium and lead.

The removal of toxic ions by membranes is more likely to depend on the size exclusion mechanism. Adsorption may contribute to the rejection of metal ions due to weak molecular forces, hydrogen interactions, and ionic attraction [86].

The concentration of the feed solution used in the research is set at 500 parts per million, and as the feed concentration increases, the percentage rejection decreases.

The reduced availability of several active adsorption sites is the reason behind this [13, 87]. A comparative statistical rejection study is presented in Table 3.

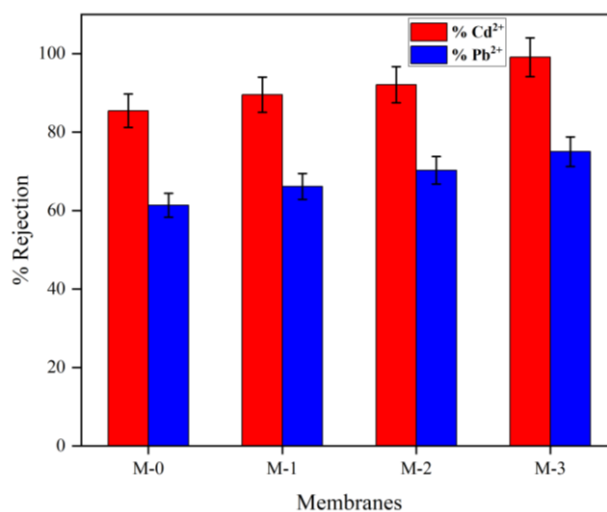


Fig. 8. Heavy metal rejection studies of membranes

Table 2. Membrane properties

Membranes	PWFL/m ² /h	Water uptake (%)	Porosity (%)	FRR (%)	Rt (%)	Rrev (%)	Rirr (%)
M-0	44.57	51.79	193.07	81.57	40.64	8.32	18.42
M-1	99.75	69.72	198.13	88.13	24.2	9.57	11.86
M-2	89.14	69.47	220.44	89.27	19.14	12.33	9.57
M-3	202.68	73.72	337.34	90.42	19.04	22.51	10.72

Table 3. Comparison of membrane performance with other reported polysulfone membranes

Entry	Polymer	Nanofiller	Operating conditions	Heavy metal	Feed (ppm)	Removal (%)	Reference
1	Polysulfone	Sulfonated polyimide	4.0 bar, Dead-end filtration	Lead and Cadmium	500	97.6 and 92.2	[88]
2	Polysulfone	Titania nanotubes and magnetite oxide hybrid nanoparticles	Cross-flow filtration	Lead and Cadmium	-	98 and 98	[89]
3	Polysulfone	MOF-5@GO	2.0 bar, Cross-flow filtration	Lead and Cadmium	-	99 and 99	[90]
4	Polysulfone	GO, ZnO, NiO	2.0 bar	Lead and Cadmium	200	30.81 and 35.48	[91]
5	Polysulfone	Mesoporous Mg-Al-Ti ternary composite oxide	-	Lead and Cadmium	-	90 and 90	[92]
6	Polysulfone	Multiwalled carbon nanotubes and polyethylenimine	0.2 bar, Dead-end filtration	Lead and Cadmium	-	96.2 and 93.87	[93]
7	Polysulfone	Multiwall carbon nanotubes and chitosan	6.0 bar, Dead-end filtration	Lead	10	71.3	[94]
8	Polysulfone	Jute copper nanocomposite	-	Lead and Cadmium	1	85.2 and 84.3	[95]
9	Polysulfone	montmorillonite	1.0 bar dead end	Lead and Cadmium	5	3 and 12	[96]
10	Polysulfone	sodium dodecyl sulfate	2.0 bar, Dead-end filtration	Lead	50	90.52	[97]
11	Polysulfone	Carbon nitride	RO	Lead and Cadmium	-	95 and 80	[98]
12	Polysulfone	Al-Ti ₂ O ₆	RO	Lead and Cadmium	-	99 and 98	[99]
13	Polyether sulfone	-	Cross-flow filtration	Lead and Cadmium	10	99 and 78	[87]
14	Chitosan-Polysulfone	Dendritic mesoporous silica nanoparticles	Dead-end filtration	Lead and Cadmium	20	88 and 97	[100]
15	Polysulfone	NH ₂ -MIL-125	2.0 bar, Dead-end filtration	Lead and Cadmium	500	75.02 and 99.10	In this study

4. Conclusion

The novelty of this study lies in the use of the amino-functionalized metal-organic framework, NH₂-MIL-125(Ti), as a filler in polysulfone mixed matrix membranes for heavy metal removal. While our earlier work employed the parent MIL-125(Ti) MOF for dyes (RB-5, RO-16) and metal ions (Pb²⁺, Cd²⁺) rejection, the present investigation focuses exclusively on Pb²⁺ and Cd²⁺ separation using the functionalized analogue.

The incorporation of -NH₂ groups not only enhanced the interaction between the MOF and the polymer matrix but also improved the affinity toward heavy metal ions, resulting in significantly higher rejection efficiencies (99.1% for Cd²⁺ and 75.02% for Pb²⁺) compared to the previously reported values (89.33% and 68.81%, respectively, for MIL-125(Ti)). This work, therefore, demonstrates the superior performance of functionalized MOFs in mixed-matrix membranes and provides new insights into the role of linker

modification in enhancing membrane-based water purification.

Acknowledgements

The writers express their gratitude to the management of NITK Surathkal, India, for the resources provided for their research. They also extend their heartfelt thanks to the Ministry of Mining, Government of India, for their financial assistance. Additionally, the authors acknowledge the support from the heads of the Department of Metallurgical & Materials Engineering and the Department of Chemical Engineering at NITK Surathkal, India, for their analytical guidance.

Author's contribution

Shivarama B. conducted the experiments and prepared the original draft of the manuscript. **Arun M. Isloor** supervised the overall research work. **Chivukula Suryanarayana Murthy** provided technical inputs and guidance. **Balakrishna Prabhu** assisted with technical analysis and assisted in carrying out selected characterization studies. All authors reviewed and approved the final manuscript.

Conflict of interest

No potential conflict of interest was reported by the authors.

Data availability

The datasets generated and/or analyzed during the current study are available from the corresponding author on reasonable request.

Funding

The authors gratefully acknowledge the financial support provided by the Ministry of Mining, Government of India, for carrying out this research.

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<https://doi.org/10.1021/acsomega.5c02486>

How to cite this paper:



Bhat, S., Isloor, A. M., Murthy, C. Sn. & Prabhu, B. (2026). High-Performance Polysulfone/NH₂-MIL-125 Membranes for the Rejection of Toxic Metals in Aqueous Solutions. *Advances in Environmental Technology*, 12(1), 103-122. DOI: 10.22104/aet.2026.7601.2133