Journal of Membrane Science & Research

journal homepage: www.msrjournal.com

Research Paper

Degree Of Crystallinity, Electrochemical Performance Studies for Desalination of Hyper Saline Water Using Polyvinyl Chloride-Based Zirconium Oxide Nanoporous Pathway for Ion Transport

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Article info

Received 2025-10-13
Revised 2026-01-17
Accepted 2026-01-28
Available online 2026-01-28

Keywords

Wet chemical method
Nanoporous pathway for ion transport
Crystalline size
Percentage crystallinity
Electrochemical characterization

Highlights

- The PVC zirconium oxide membrane was synthesized by the co-precipitation method.
- High chemical stability ensures the membrane's long-term durability.
- Semi-crystallinity enhances thermal and mechanical properties.
- The crystalline size of nano composite membrane is 24 nm.
- The membrane proved suitable for separation saline water applications.

Graphical abstract

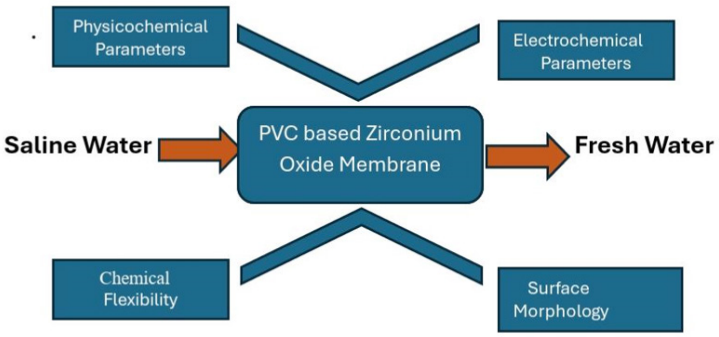


Figure – Desalination of Hypersaline water by a PVC based Zirconium Oxide, a hybrid multifunctional nanocoated membrane

Abstract

In this paper, we present an overview of an innovative approach based on the observation of chemical, surface and electrochemical properties of an inorganic membrane. The PVC based nanocoated zirconium oxide (ZO) membrane has been selected for this purpose that has been prepared by wet chemical method. The optimal performance of the PVC-based (ZO) membrane was achieved with a composition comprising 25% polyvinyl chloride (PVC) and 75% zirconium oxide. Then, this inorganic matrix enhances the electrochemical properties of a membrane like chemical, flexibility, surface morphology, mobility and many more. The preparation and application of this nanoporous pathway for ion transport membrane is greatly affected by surface, interfacial and electrochemical parameters. Crystallinity indicates the regular order of the particles in composite membrane and it relates mechanical strength, thermal stability, and ion transport. Furthermore, results of degree of crystallinity, electrochemical performance and morphology give better efficiency for desalination of hyper saline water, making it a promising candidate for treating high-salinity brines. The findings of this study serve as a foundation for future scale up using industrially compatible fabrication methods, where pressure requirements can be optimized while preserving membrane performance.

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DOI: 10.22079/jmsr.2026.2074534.1729

1. Introduction

Water is a crucial component for life to exist on Earth, water is essential, particularly in regions where fresh water is scarce. Desalination is therefore a great method of addressing the growing demand for water. Existing water supplies are under pressure as the world's population continues to rise and the demand for freshwater increases. The world's oceans have an average salinity of 3.5%, or 35 g/L. Desalination aims to bring ocean water's salinity down to levels suitable for human consumption. Traditional separation techniques have been replaced by membrane technology in recent years due to global issues with water scarcity, energy shortages, and environmental pollution. The problem is made worse by pollution, which contaminates freshwater sources and decreases their availability. Desalination is important and necessary in areas facing freshwater scarcity. By removing salts and other impurities from seawater, it provides a reliable and sustainable source of potable water for domestic, agricultural and industrial use. This approach is especially effective in dry regions where population growth is increasing and weather conditions are deteriorating.

Reverse osmosis, distillation, and electrolysis are common techniques to separate water. These methods lower the salt content and make the water better for a variety of uses. The world's problems with not enough water, not enough energy, and more pollution have led to a rise in the need for membrane technology. Because they are cost-effective and can be scaled up, these processes are in high demand [1-5].

Inorganic membrane technology, using ceramics, metals, or zeolites, offers an efficient, durable alternative to polymer membranes for desalinating hypersaline water. These membranes resist fouling, degradation, and chemical attack better, thriving in extreme conditions with higher temperatures and harsher cleaning. They also provide superior mechanical strength, permeability, and salt rejection. Using inorganic membrane technology to desalinate hypersaline water is a new method that aims to make desalination processes more efficient and long-lasting. Inorganic membranes, which are usually made of ceramics, metals, or zeolites, are better than traditional polymer-based membranes for desalination, especially when dealing with hypersaline water, which has a lot of salts and other minerals dissolved in it. Compared to organic membranes, inorganic membranes are less likely to get dirty, break down, or be attacked by chemicals. This makes them a good choice for extreme conditions like those found in hypersaline environments. They can handle hotter temperatures and harsher chemical cleaning, which makes them last longer and means you don't have to clean them as often. In addition, inorganic membranes usually have better mechanical strength, permeability, and salt rejection selectivity [6-9].

Researchers explore ceramic, silica, and titanium oxide membranes for hypersaline desalination via reverse osmosis (RO) and pressure-driven filtration, removing salts and impurities. Demand grows for options that cut costs, energy, and environmental impacts like brine discharge and chemical waste. Despite challenges in cost, scalability, and brine management, ongoing R&D shows promise for more efficient, durable systems. Researchers have looked into a number of inorganic membranes for desalination in very salty water. These include ceramic membranes, silica-based membranes, and titanium oxide membranes. These materials are often used in advanced filtration methods like reverse osmosis (RO) and pressure-driven filtration, which can get rid of salts and other impurities in the water. There is a lot of demand for new membranes because they lower costs, energy use, and environmental impact, such as brine discharge and the disposal of chemicals used in pretreatment processes. Inorganic membrane technology has a lot of potential, but there are still problems with its cost-effectiveness and scalability, as as with getting rid of brine. But research and development in this area is still going on, and the results are looking good form well desalination work better and last longer [10-12].

Inorganic membranes outperform organic ones by resisting fouling, degradation, and chemicals in hypersaline extremes. They endure higher temperatures/aggressive cleaning for longer life and less maintenance, plus offer superior mechanical strength, permeability, and salt rejection. Challenges remain in cost, scalability, and brine disposal, but R&D promises better performance and sustainability [13-16].

Inorganic membranes are more resistant to fouling, degradation, and chemical attack compared to organic membranes, making them well-suited for extreme conditions like those found in hypersaline environments. They can withstand higher temperatures and more aggressive chemical cleaning, which increases their lifespan and reduces the need for frequent maintenance. Additionally, inorganic membranes typically exhibit higher mechanical strength, better permeability, and improved selectivity for salt rejection, which makes them ideal for the high-salinity brine encountered in hypersaline desalination [17-20].

Despite their significant potential, inorganic membrane technologies continue to face challenges in cost efficiency, scalability, and sustainable brine disposal. However, ongoing research and development in this area show promising results in improving the performance and sustainability of desalination processes for hypersaline water [21-23].

Zirconium oxide membrane based on PVC is essential for desalinating hypersaline water because they improve salt rejection and water permeability. Zirconium oxide is added to the membrane to increase its mechanical strength, chemical stability, and fouling resistance, which makes it suitable for treating brine or extremely saline water. These membranes maintain high rejection rates for ions like sodium and chloride while selectively filtering out salts and other contaminants, letting freshwater flow through. It is perfect for desalinating hypersaline water because of the combination of PVC and zirconium oxide, which guarantees longevity and effectiveness even in harsh environments.

2. Experimental Section

2.1. Materials and chemicals used

Zirconium oxide (0.5 M) (Sigma-Aldrich, India), used as a binder, was mixed with polyvinyl chloride (PVC) (Otto Kemi, Mumbai, India), ground, and sieved to a 200-mesh size. This composite was then used in conjunction with various electrolytic solutions KCl, NaCl, and LiCl procured from E. Merck Limited, India. Double distilled water was used for the preparation of above compounds so, that solutions can gave better results

2.2. Nano hybrid Membrane preparation

As a binder polyvinyl chloride (otto Kemi, India, AR), was used that was grounded and sieved into 200 meshes. To make an inorganic hybrid material, mortar and pestle is used to uniformly mix Zirconium oxide with polyvinyl chloride. The membrane was prepared by heating the mixture in an oven (Oven-Universal, Memmert Type) held at 200^0C temperature for about an hour [9]. The mixture was put in a cast die, which was then moved to a pressure system (SL-89, UK), and different pressure were applied during the preparation of the membranes, including 60, 100, 140, 180, and 220 MPa. To prepared a mechanically, chemically and morphologically stable membrane, polyvinyl chloride (binder) should be 25% and inorganic material (zirconium oxide) should be 75% [24].

2.3. Properties and physicochemical performance of the nano composite membrane

The properties and physicochemical performance of nanocomposite membranes play a vital role in determining their efficiency and suitability for various applications such as filtration, desalination, and separation processes. Key characteristics include water content, thickness, swelling behaviour and mechanical strength [25].

2.3.1. Basic characterization in terms of Percentage of Water content, Thickness and Swelling

The water content is measured as the percentage of water retained in the membrane, it indicates hydrophilicity and ability to facilitate water transport. Thickness influences the membrane's permeability, with thinner membranes generally offering higher flux but reduced mechanical strength. Swelling behavior, which refers to the membrane's expansion when immersed in water, can affect its stability and performance under different conditions.

In the present section, the weight of the dry membrane was initially recorded. The membrane was then submerged in double distilled water for 7 or 9 hours at room temperature. After cleaning the surface with filter paper and removing the water molecules attached to the surface then membrane weighted again. The weight change after hydration was used to calculate the water absorption of a composite membrane. The thickness of membrane was calculated by taking the average thickness of the membrane by using screw gauze. Swelling is measured as average thickness of the membrane balanced with 1 molar univalent electrolyte for 20 hours.

The chemical stability of nanocomposite membranes refers to their resistance to degradation or chemical reactions when exposed to harsh environments, such as high temperatures, acidic or basic conditions, and organic solvents. High chemical stability ensures the membrane's long-term durability, performance, and reliability in various industrial and environmental applications. In this chapter chemical stability is determined through ASTM D543-95 method. Membrane was exposed to acidic and alkaline media as well as also allowed to react with different oxidizing agent and reducing agents and results were checked and recorded after 15, 30, 45 and 60 different properties like colour, texture, brightness, holes, bubbles and adhesiveness.

2.4 Characterization of the PVC based ZO Composite Membranes

FTIR (Fourier Transform Infrared Spectroscopy) is used to characterize nanocomposite membranes by identifying functional groups and molecular interactions. It provides insights into the chemical bonding, the presence of nanoparticles, and the interactions between the polymer matrix and incorporated nanomaterials. This helps evaluate membrane composition, structure, and surface modifications. A comprehensive set of analytical techniques was employed to characterize the structural, morphological, and microstructural features of the synthesized membranes. Fourier Transform Infrared (FT-IR) spectroscopy was carried out using a Perkin Elmer Spectrum (version 10.03.09) spectrophotometer. Samples were prepared using the KBr pellet method, and the recorded spectra were analysed to identify characteristic functional groups in the organic, inorganic, and composite components.

The crystalline structure of the materials was examined by X-ray Diffraction (XRD) using a Rigaku Miniflex diffractometer operated at 40 kV and 30 mA with Cu-Kα radiation (λ = 1.5406 Å). Diffraction patterns were collected across a specified 2θ range at a scanning speed of 6°/min to evaluate the phase composition and degree of crystallinity. To investigate the internal microstructure and nano structural features, Transmission Electron Microscopy (TEM) analysis was conducted using a JEOL 2200 system. Selected Area Electron Diffraction (SAED) and High-Resolution TEM (HRTEM) were used to visualize particle dispersion, lattice arrangements, and crystallographic orientations within the inorganic and composite phases. Surface morphology and structural uniformity were further studied by Scanning Electron Microscopy (SEM) using a JEOL JSM-6510LV instrument. SEM images revealed the distribution of fillers, membrane surface texture, and the porosity characteristics of the composite membranes [26,27].

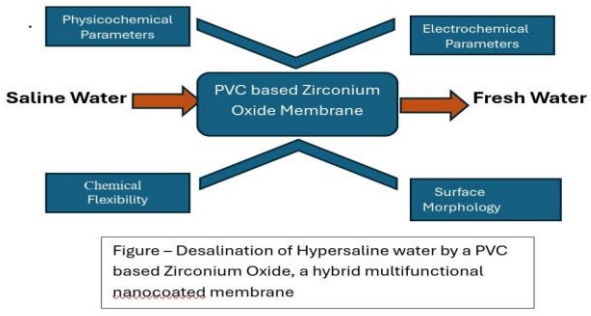


Fig.1. Desalination of hypersaline water by a PVC based ZO nano composite membrane.

3. Result and Discussion

The chemical stability of the PVC based ZO nano composite membrane was examined by immersing it in acidic, alkaline, and neutral support for 6, 12, 18, 24 and 48 hours and indicating good chemical efficiency. The membrane exhibited a water content level about 6 % and thickness (0.070 cm) was measured using a micrometer screw gauge. The swelling nature of the membrane was evaluated by submerge it in distilled water at room temperature and exhibited no observable swelling, even after prolonged immersion

FTIR spectra of the ZrO_2 and PVC/ZrO_2 nanocomposite are shown in Fig. 2. The ZrO_2 nanoparticles spectrum noticeably reveals the main characteristic sharp peak at 746 cm^{-1} that is attributed to Zr-O stretching vibrations as shown in Fig. 2 (a). In case of PVC/ZrO_2 nanocomposite, the bands of PVC can't be observed due to low loading of PVC in PVC/ZrO_2 nanocomposite [28, 29]. The peaks of Zr-O stretching in PVC/ZrO_2 nanocomposite observed at 756 cm^{-1} which slightly shifted from pure ZrO_2 nanoparticles indicating the interaction between PVC and ZrO_2 nanoparticles as shown in Fig. 2 (b).

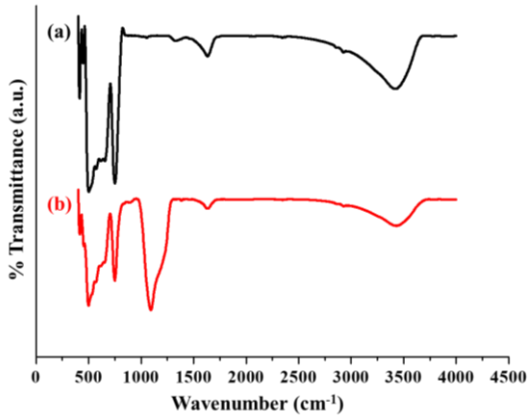


Fig. 2. FT-IR spectra of: (a) ZrO_2 nanoparticles and (b) PVC/ZrO_2 nanocomposite.

The morphology of PVC and PVC/ZrO_2 nanocomposite were studied by SEM as shown in Fig. 3. Fig. 3 (a) and Fig. 3 (b) exhibit the SEM micrographs of ZrO_2 nanoparticles which appears to be like granules and finely dispersed. The morphological differences between PVC and PVC/ZrO_2 nanocomposite are apparent at 1 and 5 µm magnifications. It may be observed that from Fig. 3 (c) and Fig. 3 (d), the PVC/ZrO_2 nanocomposite shows agglomerated morphology, which indicates that the PVC bind the surface of ZrO_2 nanoparticles [30].

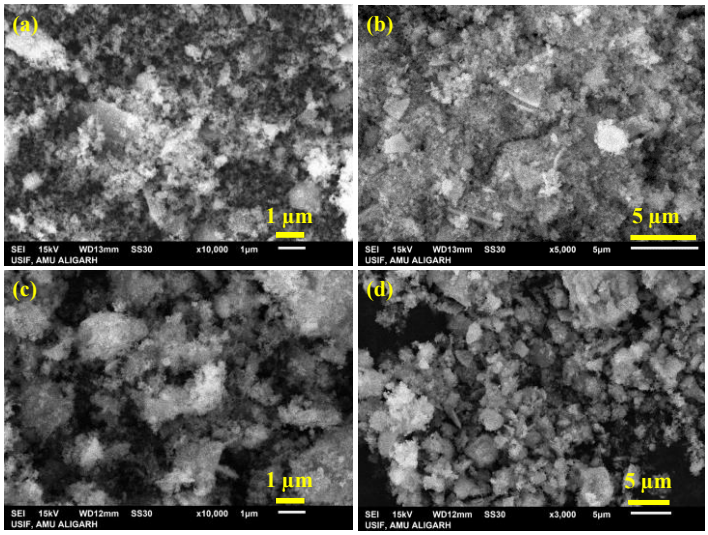


Fig. 3. SEM micrographs of: (a and b) ZrO_2 nanoparticles, (c and d) PVC/ZrO_2 nanocomposite at different magnifications.

Fig. 4 shows the TEM images of PVC and PVC/ZrO_2 nanocomposite. TEM micrograph of ZrO_2 nanoparticles reveals the irregular sized particles around 20 nm size as shown in Fig. 4 (a). The TEM images of PVC/ZrO_2 nanocomposite also reveals the same morphology as that of ZrO_2 nanoparticles but the PVC coatings at ZrO_2 nanoparticles can be shown as light grey covering around ZrO_2 nanoparticles as shown in Fig. 4 (b). By this observation it may attributed that the nanoparticles of PVC has completely mixed with ZrO_2 nanoparticles. The observed structure promote a nonuniform but interconnected pore network rather than a simple dispersed nanofiller system [31].

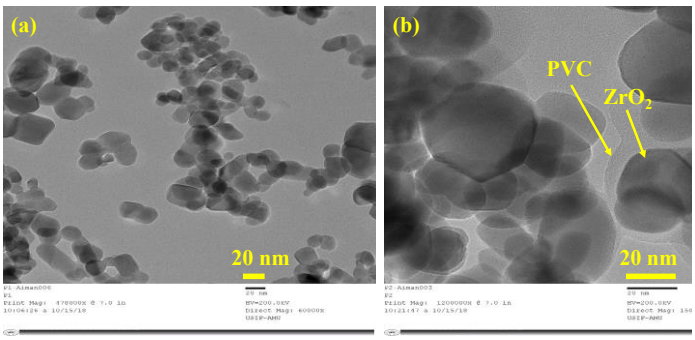


Fig. 4. TEM micrographs of: (a) ZrO_2 and (b) PVC/ZrO_2 nanocomposite.

X-Ray Diffraction Analysis

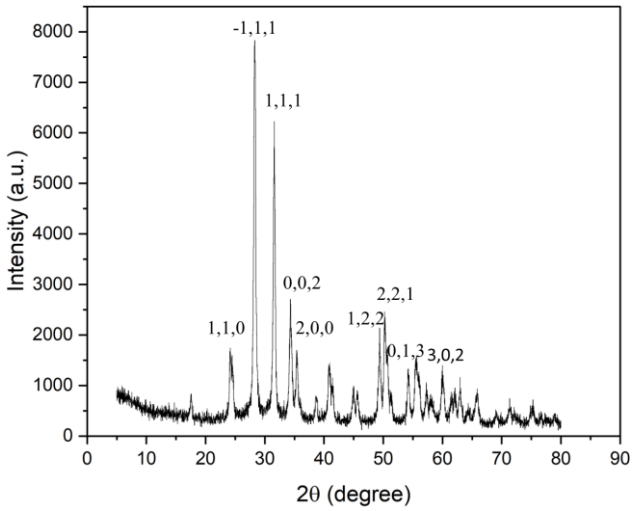


Fig. 5. XRD pattern of PVC/ZrO_2 nanocomposite.

Fig. 5 shows the X-ray diffraction pattern of PVC/ZrO_2 nanocomposite. The broad hump between 05° and 20° 2θ, indicates the amorphous nature of the nanocomposite due to the presence of PVC. The (-111) and (111) planes correspond to the most pronounced peak at approximately at 28.4° and 31.5° 2θ, which indicates the monoclinic phase of ZrO_2. Miller indices that match conventional reflections for stabilised ZrO_2, notably (1,1,1), (2,0,0), and (1,3,0), indicate the presence of tetragonal phase of crystalline ZrO_2. Additional peaks at higher angles (such 34.6°, 49.4°, and 60° 2θ) show higher-order planes, which means that there are nanoscale ZrO_2 particles that may be growing or aggregating in different ways. ZrO_2 as a crystalline filler, the nanocomposite's semi-crystalline structure raises the possibility of improvements in thermal stability and mechanical strength.

Percent Crystallinity:

The crystallinity of the PVC based ZrO_2 nano composite membrane was calculated by using the X-ray diffraction (XRD) method. This technique involves drawing a straight baseline beneath the diffraction curve and computing the relative contribution of crystalline peaks compared to the total diffracted intensity (crystalline + amorphous). The percentage crystallinity was calculated using the following relation:

For the PVC based ZrO_2 nano composite membrane material,

%Crystallinity = (Area under crystalline peaks) / (Area under all peaks) ×100

Area under crystalline peaks = 24088.50

Total area under all peaks = 33311.92

%Crystallinity = 24088.50 / 33311.92×100 = 72.31%

The PVC-based ZrO_2 nanocomposite membrane demonstrates a crystallinity of approximately good 72.31%, reflecting a high degree of structural order within the composite matrix. The increased crystallinity (72.31%) of the PVC-ZrO_2 membrane does not directly imply reduced flexibility, as mechanical behavior is also controlled by amorphous phase continuity and interfacial interactions. ZrO_2 nanoparticles promote localized crystallization while preserving chain mobility in amorphous regions, thereby maintaining membrane flexibility. This behavior is consistent with the observed microstructure, where nanoparticle-induced ordering coexists with a continuous amorphous matrix. This indicate crystallinity is expected to enhance the membrane's mechanical strength, thermal stability, and chemical stability [26].

Crystallite Size:

The crystallite size of PVC-based ZrO_2 nanocomposite membrane calculated by using Debye-Scherrer equation

$$D = \frac{k\lambda}{\beta \cos\theta}$$

where, D = average crystallite size in nanometers,

k = Shape factor (~0.9),

λ = Wavelength of Cu Kα radiations taken as 1.5406 Å or 1.5406 x 10^{-10}

β = Full width at half maximum of diffraction peaks in radians (β = 0.34728)

θ = Bragg's diffraction angle (2θ = 28.30 or θ = 14.15)

With the help of above equation, the crystalline size of nano composite membrane is 24 nm and this value indicate the nano structural properties and proper arrangement of particles [32].

4. Conclusion

On the basis of physiochemical characterization of PVC-based ZO nano composite membrane, the membrane was found to be chemically stable, low water content level, good thickness, zero swelling and excellent efficiency in different environment. A wide-ranging material property and their behavior were studies in terms of electrochemical characterization by using FTIR, SEM, TEM and XRD analysis. These powerful techniques reveal the functional group, morphology, structure, crystalline nature, percentage of crystallinity and particle size. Lastly, the aforementioned membrane has drawn interest from academics and is being used in methods for desalinating hypersaline water and separation process.

Funding Declaration

The authors are grateful to the Chairman, Department Of Chemistry AMU Aligarh for providing necessary research facilities, USIF AMU for SEM, TEM and EDX analysis and Zakir Hussain College of Engineering and Technology AMU Aligarh for XRD studies.

Declaration of Interest Statement

All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed by [Mohammad Mujahid Ali Khan], [Nazia Zeeshan], [Durgesh Wadhwa], [Salman Siddiqui], [Sharique Ahmad] and [Syyed Asad Ali]. All authors read and approved the final manuscript.

Data availability

The data supporting this study's findings will be made available from the corresponding author upon reasonable request.

Credit authorship contribution

N. Zeeshan: Conceptualization, funding acquisition, Project administration, Supervision, Writing-review & editing.
M. M. A. Khan: Conceptualization, formal analysis, Methodology Supervision.
D. Wadhwa: Conceptualization, Supervision, formal analysis, methodology.
S. Siddiquid: Data curation, Investigation, Methodology, project administration; resources, supervision, writing-review and editing.
S. Ahmad: Data curation, Investigation, Methodology, project administration; writing-review and editing.
S. A. Ali: Investigation, data curation, supervision, resources.
A. Rehman: Data curation, formal analysis, investigation methodology, Writing-original draft, Writing- review and editing.

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