

Fabrication of highly effective aramid fiber-based polypropylene composite membranes for desalination

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Abstract—Water is essential for human survival and existence. Although everyone acknowledges the importance of water reserves, increasing industrialization and other human activities are drastically reducing the quality of the water. Polymeric composite membranes provide a dependable answer for removing water pollutants and thus improving its quality. In this study, polypropylene composite membranes with Aramid support (PM1 membranes) were fabricated using thermally induced phase separation (TIPS) for water purification and characterized using SEM. Numerous membrane characteristics, including polymer concentration, oil for dissolving polymer, nucleating agent, water immersion time and organic solvent were all adjusted for the production of extremely efficient PM1 membranes. The PM1-WC membrane, which was the best membrane, was fabricated by combining 0.1 g PP with soyabean oil, adipic acid as a nucleating agent, and n-hexane as an organic solvent (PP membranes submerged in water for 40 minutes). In a water filtering system developed in the lab, the PM1 membranes' desalination capacities were tested. The desalination capacity of the PP membranes showed a salt rejection of between 70% and 75%.

Keywords: Separation Process, Polypropylene, Wastewater, Membranes, Desalination

INTRODUCTION

Human existence and survival depend on the availability of water. Despite the fact that everyone agrees that water reserves are crucial, rising industrialization and other human activities are seriously lowering the quality of the water. Pharmaceutical wastes, pesticides, dyes, and other commercial wastes are examples of persistent contaminants that cannot be removed by conventional water filtration techniques like sedimentation and biochemical degradation. A number of procedures are used in traditional water treatment, such as primary treatment, which use macro-filtration to remove suspended material, and secondary treatment, which employs oxidation to eliminate biological matter. Then water is subjected to tertiary treatment, which involves chemical disinfection using a number of substances, including chlorine, chlorine dioxide, ozone, potassium permanganate, and chlorine. Additionally, UV radiation is also commonly used for this purpose. Water is micro-filtered after treatment is complete before being released into the environment [1].

Wastewater can be treated efficiently, easily, and efficiently with membrane technology. It can be used to treat acidic or alkaline wastewater, separate oil from water, desalinate seawater, and remove heavy metals, etc. Membrane distillation (MD) stands out from other mem-

brane processes because it has low operating costs, is capable of rejecting nonvolatile components and inorganic ions in 100% of cases, and is not limited by the concentration of pollutants [2].

Today water purification is considered to be an industrial activity and many initiatives have been taken to guarantee the availability of clean water. Typically, membranes are used to filter the water after it has been purified. Membranes have made it possible to purify against a very large collection of pollutants. Polymeric membranes provide a low-energy, affordable, and effective way to purify water. It is, however, important to develop a less expensive and more efficient technology for wastewater purification in light of the financial and social factors [3].

Membrane material is frequently preferred due to its desirable thermal and mechanical properties as well as its chemical stability. In the formation of the porous supports layer and the composite membrane active layers, a variety of polymers are used, including polyvinyl chloride (PVC), polyacrylonitrile (PAN), poly-methacrylic acid (PMAA), polyvinyl alcohol (PVA), polypropylene (PP), polyethylene Glycol (PEG) and polypropylene (PP) [4-8]. A polymeric membrane's porous design typically performs two functions: mechanical support (as in composite microfiltration or ultrafiltration membranes) or as a useful separating layer in those membranes. Mechanical steadiness in membranes is contingent upon the properties of the porous membrane, the membrane substance, and the porous design. When an external force causes compaction of the membrane, it may undergo significant distortions, resulting in strik-

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ing alterations under pressure, such as decreased solvent permeability and lower performance with greater retention of solutes. To ensure mechanically stable membranes, it is preferred to have high anti-compaction qualities. This necessitates research on how porous nature of materials affect the performance of the composite membranes for water purification.

Desalination has also been done with polyether sulfone membranes that have been phase-inverted [9]. These PES membranes have a flow rate of $150 \text{ L } \mu\text{m m}^{-2} \text{ h}^{-1}/\text{bar}$ after being augmented with silica NPs. Transmission electron microscopy (TEM), scanning electron microscopy (SEM), thermal gravimetric analysis (TGA) and Fourier transform infrared (FTIR) spectroscopy have been used to evaluate the morphology and chemistry of these membranes [10].

Polymeric membranes can be fabricated by a variety of procedures. Among those, phase inversion secures a prime spot owing to its feasibility and simpler synthetic steps. In the past, there have been four main categories of phase inversion techniques: Vapor-induced phase separation (VIPS), thermally induced phase inversion (TIPS), non-solvent induced phase inversion (NIPS) or immersion precipitation and solvent evaporation-induced phase separation (SEIPS). High temperatures are used to create the TIPS polymer solution, which is then cooled to cause polymer solidification and phase separation [11]. With the aid of a casting dye, the NIPS technique fabricates the required membrane out of a homogeneous solution of the polymeric matrix and a non-solvent [12].

TIPS can be utilized to create a dual-layer nanocomposite membrane using PES (Polyether sulfone) and PVDF (Polyvinylidene fluoride) for the treatment of wastewater from petrochemical refineries. Multiwalled carbon nanotubes (MWCNTs) have been integrated into the composite membrane system; these membranes have antibacterial qualities and are hydrophilic and porous in nature. These membranes remove extracellular polymeric substance (EPS) with a 98.2% efficiency [13].

Unaltered PP membranes, with an average molecular weight of 14,600, have been fabricated with TIPS and a solute rejection of 95% for lysozyme was attained. These membranes have a hydrophilic character and are quite porous [14]. Additionally, it has been claimed that TIPS can be used to prepare PP coated chitosan membranes. This membrane's rejection rate is 44% at a minuscule flow rate, or $43.42 \text{ L } \mu\text{m/m}^2 \text{ h}/\text{bar}$ [15]. TIPS was used to construct PP composite membranes atop an aramid support layer in this study; operational variables were tuned and the best feasible conditions for desalination by these membranes were identified.

MATERIALS AND METHODS

Merck provided isotactic polypropylene (PP), Mw 12,000, average Mn 5,000. Qingdao Chemical Reagent Co. Ltd. in China supplied the graphite flakes. Adipic acid with 99.5% purity, benzoic acid with 99.0% purity, cinnamic acid with 99.0% purity, sodium polyacrylate with 99.0% purity and sodium chloride with 98.0% purity were purchased from Sigma Aldrich. 6% hydrogen peroxide was purchased from Sarco Chemical Industries. Mustard oil, coconut oil and soy oil were purchased from a local market. Reagent grade solvents, i.e., dichloromethane (DCM), *n*-hexane, aniline, petroleum ether, ethanol, and chloroform, were purchased from Sigma-Aldrich

and used without further purification. In all of the studies, distilled water or, in some circumstances deionized water, was used.

1. Fabrication of PP@ Aramid Composite Membranes

The membranes were fabricated by continuous stirring at 450 rpm while heating PP in the oil to 300 °C. The PP was put in various amounts to 20 mL of soyabean oil that had been placed in a stainless-steel basin. The solution was then heated again at 300 °C with the addition of nucleating agents. The 4×4 cm piece of Aramid (Kevlar) cloth, which acted as the membrane support, was immersed in this hot oil solution once the polymer was completely dissolved. It was then immediately removed and immersed in distilled water.

The membrane was allowed to stay in distilled water before being immersed for 12 hours in an organic solvent. As a result, the oil from the membrane matrix could be totally eliminated so the membrane could then finish drying. The desalination, degradation, and disinfection capabilities of the produced membranes were subsequently evaluated. For oil optimization, soybean oil, coconut oil, and mustard oil were utilized to prepare the PM1-OS, PM1-OC, and PM1-OM membranes, respectively. These designations (PM1-OS, PM1-OC, and PM1-OM) were assigned to the membranes prepared using soybean, coconut, and mustard oil, respectively. For the purpose of optimizing the polymer concentration, 19 distinct membranes with PP concentrations of 0.01, 0.02, 0.03, 0.04, 0.05, 0.06, 0.07, 0.08, 0.09, 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, and 0.9,1 grams were constructed. These membranes were designated as PM1-A, PM1-B, PM1-C, PM1-D, PM1-E, PM1-F, PM1-G, PM1-H, PM1-I, PM1-J, PM1-K, PM1-L, PM1-M, PM1-N, PM1-O, PM1-P, PM1-Q, PM1-R and PM1-S respectively.

Various nucleating agents were employed for the nucleating agent's optimization. The membranes were made by independently utilizing adipic acid, benzoic acid or cinnamic acid and the finished membranes were designated PM1-NA, PM1-NB, and PM1-NC.

Different solvents, including *n*-hexane, benzene, toluene, aniline, carbon tetrachloride, and petroleum ether were employed to optimize the organic solvent; the resulting membranes were designated as PM1-SH, PM1-SB, PM1-ST, PM1-SA, PM1-SC, and PM1-SP respectively.

Additionally, the time duration, for which the membrane was submerged in water, was also optimized. Various membranes, designated as PM1-WA, PM1-WB, PM1-WC, PM1-WD and PM1-WE, were submerged in water for 10, 20, 30, 40 and 50 minutes respectively. The prepared membranes had a thickness of 10.001 mm, according to the vernier calipers. The membranes made with different polymer concentrations showed a negligible change in thickness. Fig. 1 depicts the schematic representation of the synthesis and fabrication of PM1 membranes.

2. Characterization of Membranes

The surface morphology of several membranes was investigated using SEM analysis (JEOL 5410). Using image J software, the SEM images were also used to estimate the membranes' pore sizes.

3. Evaluation of Desalination Ability of Membranes

Experiments on desalination were carried out using manufactured membranes. The bench top cross flow filtration apparatus was used for the experiment (Fig. 2). The filtering system uses a cross-flow design and has sintered discs and adjustable membrane cells

desalination for some of the membranes.

$$\% \text{ desalination} = \frac{EC_i - EC_f}{EC_i} \times 100$$

where EC_i is EC of feed solution and EC_f is EC of permeate solution.

RESULTS AND DISCUSSION

SEM was used to examine the surface morphology of PM1 series membranes. First, the support layer of a blank Aramid fabric was examined. Fig. 3 displays the SEM images of blank membranes.

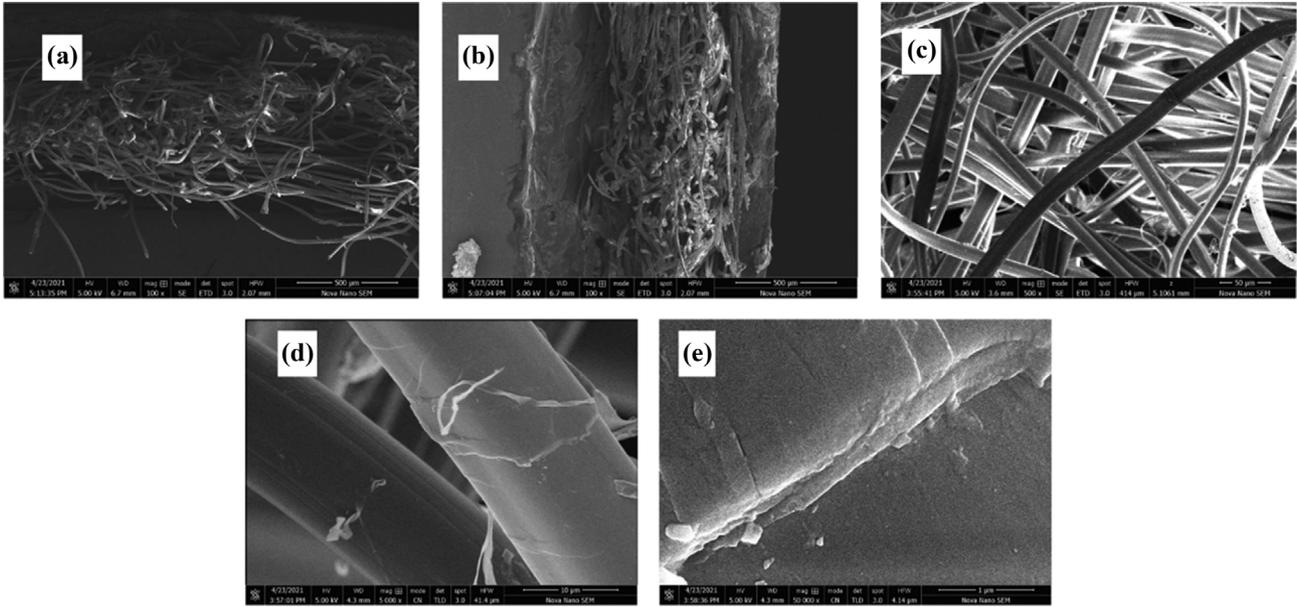


Fig. 3. (a & b) 100x magnification cross-sectional analysis of a blank membrane, (c) 500x magnification cross-sectional analysis of a blank membrane, (d) 5000x magnification surface morphological analysis of a blank membrane, and (e) 50000x magnification surface morphological analysis of a blank membrane (working distance (WD) 4.3 mm).

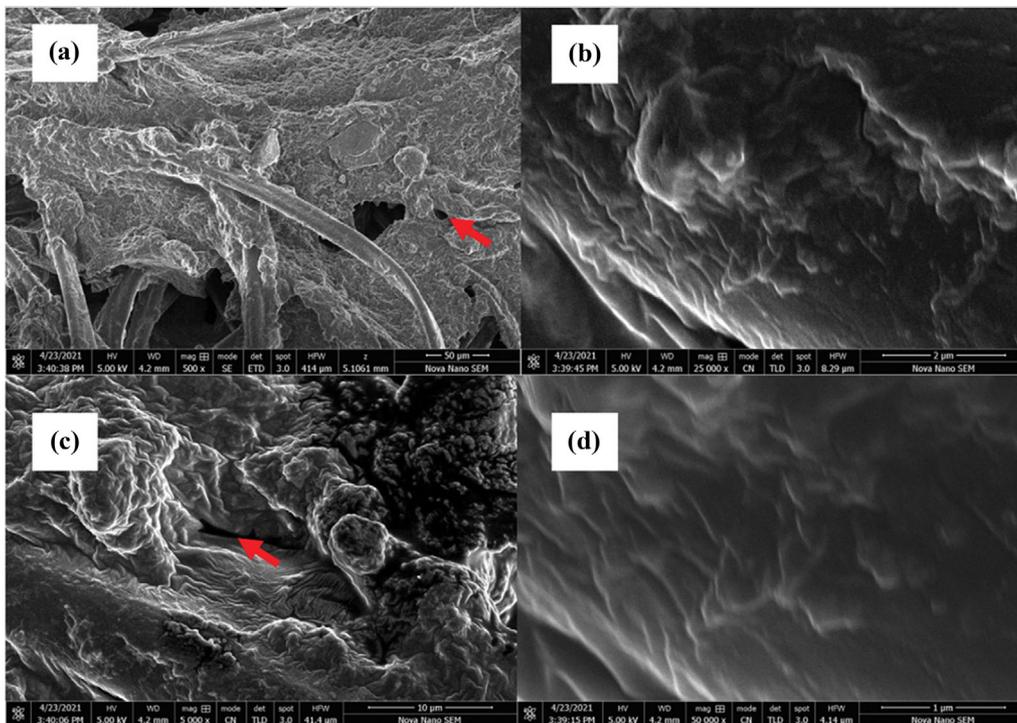


Fig. 4. (a) 500x magnification cross-sectional study of the PM1-I membrane, (b) 25000x magnification surface morphological analysis, (c) 5000x magnification surface morphological analysis, and (d) 50000x magnification surface morphological analysis. PM1-I is a membrane made entirely of PP with a 0.09-gram PP covering,

Using an electron accelerating voltage of 5.0 kV, HFWs range between 2.07 mm and 414 mm, magnifications of 100, 500 and 50 k were used to capture images of blank membranes. An image of

the membrane that was taken at a lower magnification shows the support layer's fibers Fig. 3(a & b), 3(c).

SEM photographs of PM1-I polymeric membranes are exhib-

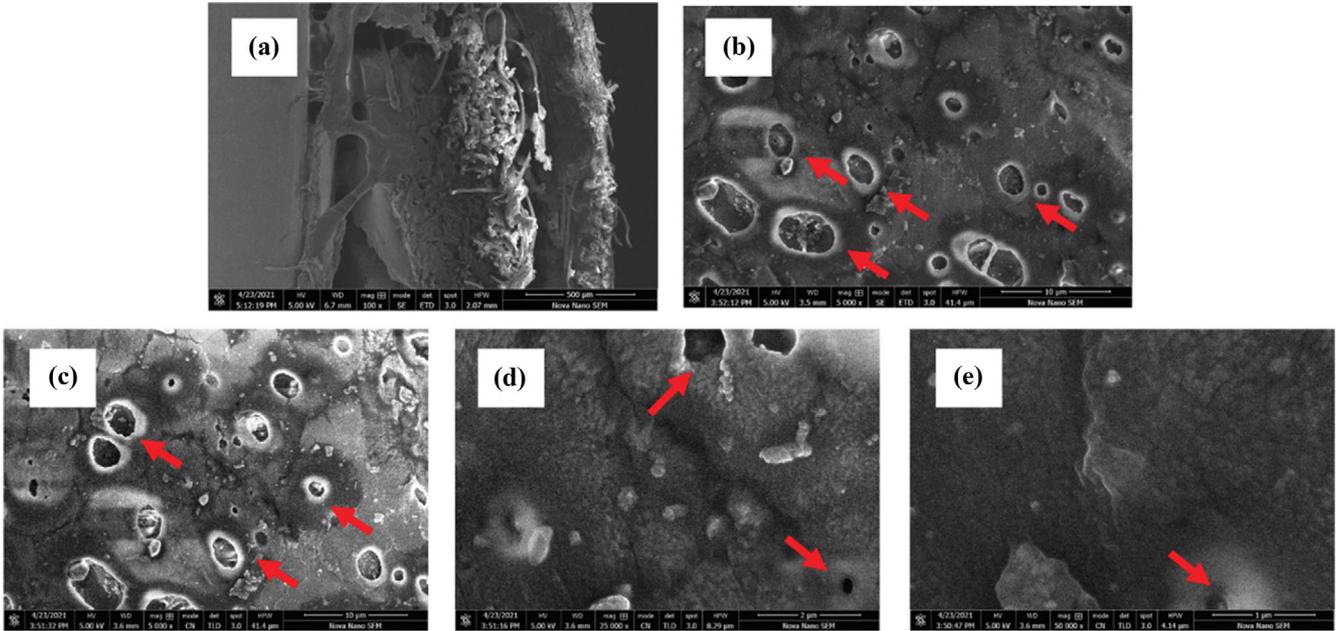


Fig. 5. (a) Shows a cross-sectional analysis of the PM1-J membrane magnified 100 times; (b) shows a surface morphological analysis of the PM1-J magnified 5000 times (WD=3.5 mm); (c) shows a surface morphological analysis of the PM1-J magnified 5000 times (WD=3.6 mm); (d) shows a surface morphological analysis of the PM1-J magnified 25000 times; and (e) shows a surface morphological analysis of the PM1-J.

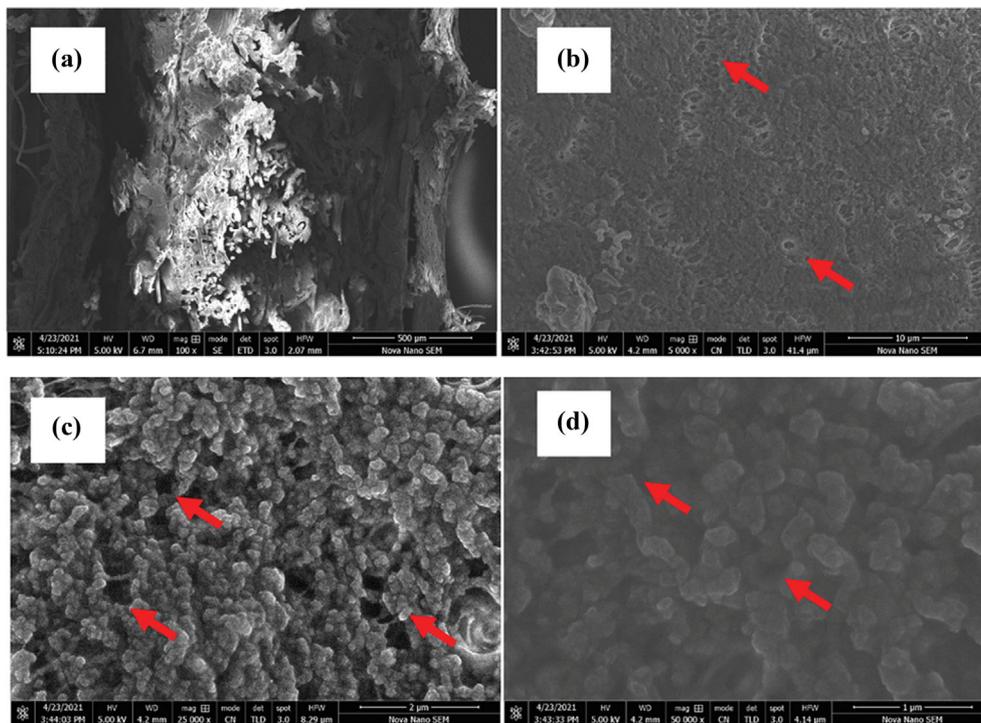


Fig. 6. PM1-K membrane cross-sectional analysis at 100x magnification is shown in (a), (b), (c), and (d). (a) Surface morphology examination of PM1-K at 5000x magnification, 25000x magnification, and 50000x magnification. (b) Surface morphology examination of PM1-K at 5000x magnification. where PM1-K is a membrane made entirely of PP with a 0.2 gramme PP covering.

ited in Fig. 4. Images of the PM1-I (Pristine PP membrane coated with 0.09-gram PP) membrane were obtained at magnifications of 100, 500, 25,000, and 50,000 at an acceleration voltage of 5.0 kV for the electrons and HFWs of 41.4 m and 8.20 m at various magnifications. Fig. 4(b), 4(c) and 4(d) clearly show the polymer's smooth surface over the aramid fiber. Red marks serve as indicators for the membrane holes that may be seen in the photos.

Fig. 5 shows SEM images of PM1-J membranes (pristine PP polymeric matrix coated with 0.1-gram PP). The SEM image of the PM1-I membrane was acquired at magnifications of 100, 5000, 25000, and 50000 with HFWs of 41.4 m, 8.90 m, and 2.07 mm for various magnifications at an accelerating voltage of 5.0 kV for the electrons. The SEM images of the polymeric matrix clearly show the pores in composite polymer membrane. It is evident from the comparison of the SEM images of the PM1-I membrane with those of the control polymeric matrix that the number of pores grows as the proportion of PP increases.

The SEM images of PM1-K (pristine PP polymeric matrix coated with 0.2 g PP) polymeric membranes are shown in Fig. 6. The SEM image of the PM1-K polymeric matrix was obtained at magnifications of 100, 5k, 25k, and 50k with HFWs of 41.4 m, 8.29 m, and 2.07 mm for various magnifications at an accelerating voltage of 5.0 kV for the electrons. The SEM micrographs of the membrane clearly show the pore in polymeric membrane. The membranes' porous structure is shown by the tiny pores indicated by the red arrows. Other PM1 series membranes share the same production process and general structural similarities, but because each PM1 membrane's polymer (PP) concentration varied, their SEM images are not shown.

1. Estimation of Pore Size of PP Membranes

The PP membrane pores are of the same size as those found in MF membranes; however, the hybrid membranes feature three layers, including an aramid fiber middle layer that works as a composite membrane, polymer serves as top and bottom covers and the fabric layer is sandwiched between them. The three layers' criss-crossing design gives the membrane the ability to filter in the NF

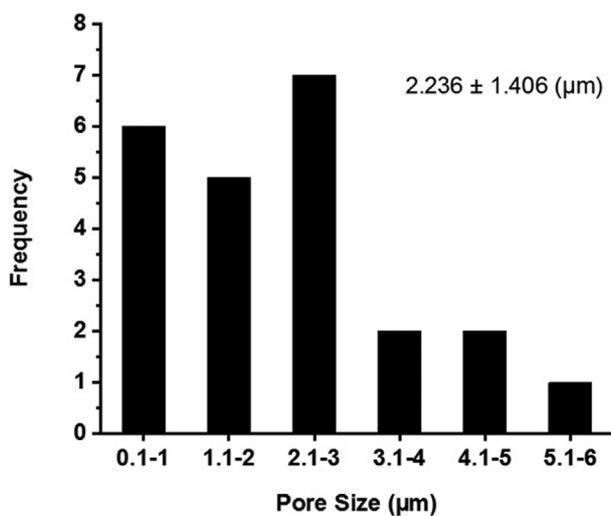


Fig. 7. The PP membranes' pore sizes as determined by Image J from SEM images.

range. Similar outcomes have been reported by [16]. By adjusting different factors like the polymer concentration, solvent type, and membrane fabrication process, it is possible to fairly regulate the pore size of composite membranes. Additionally, different additives can be used to change the membrane's pore size and distribution, including pore-forming agents and surfactants. In general, depending on the manufacturing process and the intended use, the pore size of composite membranes can range from nanometers to micrometers. The result is shown in Fig. 7. The aperture size of these membranes was estimated through the ImageJ software by the use of SEM images.

2. Evaluation of Desalination Ability of PP@ Aramid Membranes

All of the manufactured membranes were put to the test in a lab-built water filtration apparatus. For the development of highly efficient PM1 membranes, many membrane properties, including the oil for dissolution of polymer, nucleating agent, non-aqueous organic solvent, concentration of polymer and water immersion period, were optimized. By evaluating the manufactured membranes, i.e., PM1-OS, PM1-OC, and PM1-OM (where PM1-OS, PM1-OC, and PM1-OM are pure PP polymeric membranes fabricated with soyabean oil, coconut oil, and mustard oil, respectively) using filtration units one at a time, the amount of oil for dissolving PP was optimized. NaCl solution of 400 ppm was utilized for the experiment. By comparing the TDS and EC values of the feed solution before and after, the performance of the membrane was assessed. The flow meter was set on the filtration unit's control panel, the ingress of the membranes was also calculated. The membrane showed a 12 LPM flux. At 30 °C, the membrane made via PP dissolution in soybean oil (PM1-OS) had the highest desalination performance as shown in Fig. 8.

The composite membrane structure's PP polymeric layer and Aramid support layer are connected by a physical and mechanical interlocking mechanism. The PP polymeric layer fills the gaps between the Aramid fibers to form a continuous and dense membrane layer and the Aramid fibers act as a strong and flexible support structure for the PP polymeric layer. The concentration of PP

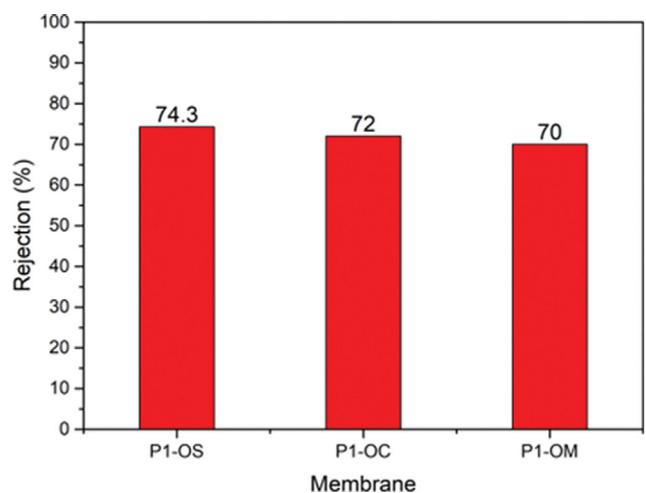


Fig. 8. Optimization of different oil for the preparation of membranes.

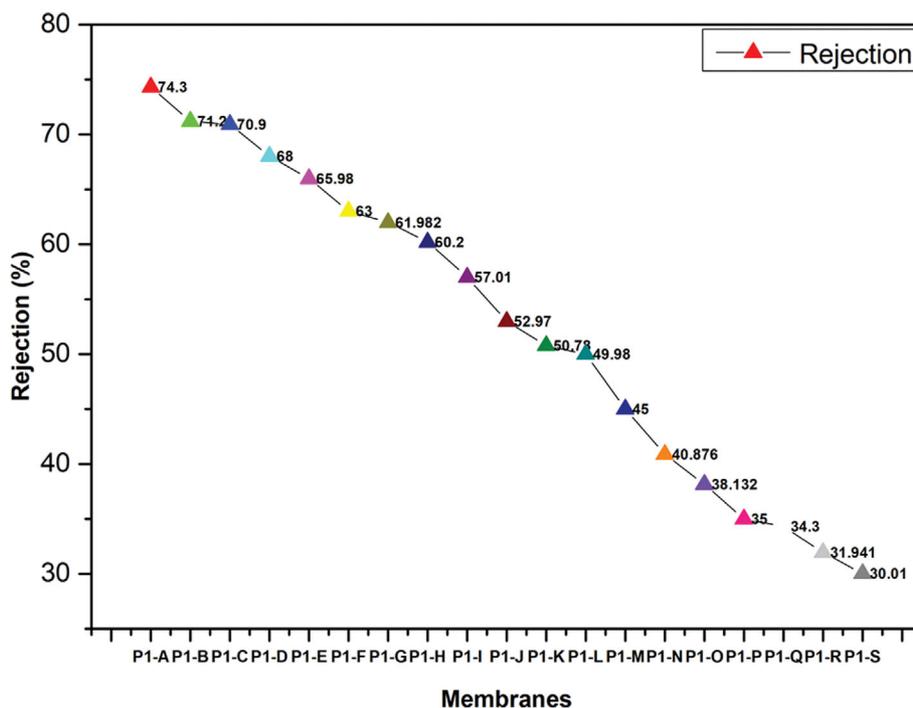


Fig. 9. Optimization of polymer concentration for the fabrication of the PP@Aramid membranes.

was optimized by testing each of the manufactured membranes with a filtration unit (PM1-A, PM1-B, PM1-C, PM1-D, PM1-E, PM1-F, PM1-G, PM1-H, PM1-I, PM1-J, PM1-K, PM1-L, PM1-M, PM1-N, PM1-O, PM1-P, PM1-Q, PM1-R and P NaCl solution of 400 ppm was utilized for the experiment). By comparing the TDS and EC values of the feed solution before and after, the performance of the membrane was assessed. The PP was dissolved in soyabean oil to create all of these membranes because it performed the best in earlier tests. The membrane made with 0.1 gm of PP (PM1-A) performed the best as shown in Fig. 9. The influx of the membranes was calculated using the flow meter mounted on the front panel of the filtration unit. Inflow rates of 12 LPM were seen on the membrane with the least amount of PP, while inflow rates of 6 LPM were noticed on the membrane with the most amount of the polymer. The constant drop in the influx rate is caused by an increase in polymer concentration. These findings can be associated with the fact that with the increase in polymer concentration, the mechanical strength of the membrane may also increase, but the permeability may decrease. This can result in reduced water flux and lower overall membrane performance. Therefore, to optimize the performance of composite membranes, it is important to carefully balance the concentration of the polymer with other fabrication parameters such as the choice of solvent, the addition of nucleating agents or other additives, and the post-treatment of the membrane. This can help to achieve a desirable balance between membrane permeability and selectivity.

For the fabrication of composite membranes, a nucleating agent was incorporated into the polymer solution to encourage the growth of uniformly sized small crystals. As a result, the membrane's mechanical and separation qualities were enhanced. To find the optimal nucleating agent, a filtering unit was used to examine the produced

membranes PM1-NA (pristine PP polymeric membranes manufactured by the addition of adipic acid), PM1-NB (pristine PP polymeric membranes manufactured by the addition of benzoic acid), and PM1-NC (pristine PP polymeric membranes manufactured by the addition of cinnamic acid). A 400 ppm NaCl solution was used for the experiment. By contrasting the TDS and EC values of the feed solution before and after, the performance of the membrane was assessed. Since it performed well in earlier tests, 0.1 gm of PP was dissolved in soyabean oil to create all of these membranes. The best performance was displayed by the membrane using adipic acid (PM1-NA) as shown in Fig. 10. Membranes PM1-NA, PM1-NB, and PM1-NC showed a 12 LPM flux. The chemical bonding between the nucleating agent and the polymer is caused by

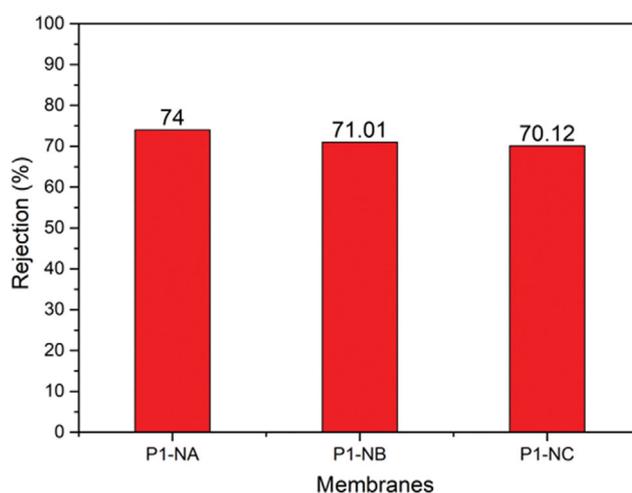


Fig. 10. Optimization of nucleating agents.

intermolecular forces such as hydrogen bonding. The precise nature of the interactions depends on the chemical properties of the nucleating agent and the polymer [17]. Here the membranes described in the study used organic acid as the nucleating agent with PP as the polymer. Adipic acid contains carboxylic acid groups (-COOH) which can form hydrogen bonds with the polymer chains in the PP solution. This promotes the formation of crystal nuclei and leads to the formation of a more uniform membrane structure.

By comparing the desalination performance of the polymeric membranes PM1-SH (pristine polymeric PP membranes immersed in n-hexane for oil removal), PM1-SB (pristine polymeric PP membranes immersed in benzene for oil removal), PM1-ST (pristine polymeric PP membranes immersed in toluene for oil removal), PM1-SA (pristine polymeric PP membranes immersed in aniline for oil removal), PM1-SC (pristine polymeric PP membranes immersed in carbon tetrachloride for oil removal) and PM1-SP (pristine polymeric PP membranes immersed in petroleum ether) for oil removal. Using NaCl solution of 400 ppm, the effectiveness of the polymeric membrane was evaluated by comparing the EC and TDS values of the feed solution prior to and after filtration through the polymeric membranes. Adipic acid was used as the nucleating agent and 0.1 g of polypropylene was dissolved in soyabean oil to create all of these membranes because it performed the best in earlier tests. The best performance was displayed by the adipic acid-prepared membrane (PM1-SH) as shown in Fig. 11.

In order to determine the exact time duration to immerse a membrane in water to achieve the best performance, the following membranes were tested with filtration units: PM1-WA (10 minutes of submersion of PP polymeric membranes), PM1-WB (20 minutes of submersion of PP polymeric membranes), PM1-WC (30 minutes of submersion of PP polymeric membranes), PM1-WD (40 minutes of submersion of PP polymeric membranes), and PM1-WE (50 minutes of submersion of PP polymeric membranes) (60 minutes of submersion of PP polymeric membranes). A 400 ppm NaCl solution was used for the desalination experiment And the performance of the polymeric membrane was evaluated by comparing the EC and TDS values of the feed before and after filtration. All of these membranes were made using 0.1 gm of PP polymer dis-

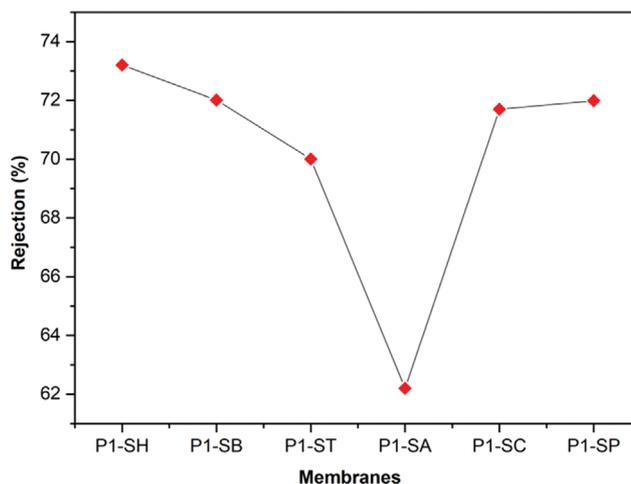


Fig. 11. Optimization of solvent for fabrication of membranes.

solved in soyabean oil, for nucleating agent adipic acid was used and n-hexane as the oil removal solvent because the membrane prepared with these parameters performed the best in earlier studies. The duration of immersion had no effect on the membrane's desalination (10 and 20 minutes, respectively). The PM1-WC, PM1-WD, and PM1-WE membranes all performed equally well during the desalination procedure, proving that 30 minutes of immersion time is adequate because a longer immersion time has little to no effect on the membrane's desalination capacity.

The PM1-WC (membrane fabricated by 40 minutes of submersion in water) was the most optimized polymeric membrane created by using soyabean oil for dissolution of PP (0.1 gm), for nucleating agent adipic acid was used and n-hexane was employed for the removal of oil as a solvent. The membrane was submerged in water for 30 minutes because previous experiments indicated that these conditions produced the best results. The PM1-WC membrane was then tested once more using a 400-ppm solution of NaCl and Na₂SO₄. When the salt solution was filtered, the membrane showed rejection rates of 74% and 72% for NaCl and Na₂SO₄ respectively.

Table 1. A comparison of various supported polymeric membranes

Polymers/support layers	Method of Fabrication	Characterization	Porous/ Non-porous	Nature	Desalination	Ref.
Psf	Phase inversion	ATR-IR XRD	porous	hydrophilic	72% Rejection	[18]
PVDF+PVA/Psf	Phase inversion	SEM AFM	porous	hydrophilic	57%	[19]
Poly(N,N-dimethylamino-2-ethylmethacrylate) Psf	Phase inversion	XPS SEM AFM	porous	hydrophilic	57.6%	[20]
Psf/Glass fiber membrane	Phase inversion	XPS SEM FTIR	Porous		58%	[21]
PP/Aramid	Phase inversion	SEM	porous	hydrophilic	<75%	Current study

3. Comparative Analysis

A comparison of various supported polymeric membranes was performed and presented in Table 1.

Based on the tabulated data, it was observed that the membranes fabricated in this study are superior to previously reported works. The prepared membranes are simple to fabricate, inexpensive, and easier to handle. They offer an outstanding solution to the current water crisis. The system's transition to industry is also a simple process that requires no complex instrumentation. The researchers advocate the use and investigation of these membranes in water purification applications.

CONCLUSION

Aramid supported PP composite membranes (PM1 membranes) were fabricated for the purpose of water purification. The membranes were fabricated via TIPS and characterized with SEM. Numerous membrane characteristics, including the oil for dissolving polymer, polymer concentration, nucleating agent, organic solvent, and water immersion time, were all optimized for the production of highly efficient PM1 membranes. The PM1-WC membrane, which was the best membrane, was created by taking soyabean oil for dissolution of PP (0.1 gm) with adipic acid as a nucleating agent and n-hexane was employed for the removal of oil as a solvent, and the membranes were submerged in water for 30 minutes for desalination experiment. The desalination capability of the PM1 membranes was assessed in a water filtration device created in the lab. The PP membranes that were tested for their desalination capacity displayed a salt rejection of between 70% and 75%.

FUTURE ASPECTS

Membrane technology has a great future because society continues to need the development of effective and efficient air/water purification techniques. This study was conducted to evaluate PP composite membranes supported by Aramid for desalination and water purification that yielded very encouraging results. It is possible that these membranes could be improved for even higher efficiency and selectivity. The PM1 membranes could be used in a variety of settings, such as industrial water treatment facilities and home water filtration systems, in terms of practical applications. As populations increase and freshwater resources become more scarce, it will be crucial to develop more effective and affordable water purification techniques. To identify the ideal circumstances for membrane fabrication, additional research may examine the use of various nucleating agents, solvents, and immersion times. To guarantee their performance over lengthy periods of use, the PM1 membranes' long-term stability and durability could also be tested. In general, the creation of PP composite membranes supported by Aramid for water filtration represents a significant development in the field of membrane technology and has the potential to deliver clean, safe water to communities all over the world.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

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