Improvement of Water Flux with Date Palm Reinforcement in Composite Membranes

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Abstract

Low energy consumption is a major reason for membrane technology's popularity. Among its many uses are water treatment, oil filtration, food and beverage industry, biomedical applications, etc. World leaders are grappling with the problem of drinking water shortages at the moment. Clean, safe water is a fundamental right of every individual. Water treatment uses membranes to separate contaminants by their size or charge. Water treatment often uses membranes, such as microfiltration, ultrafiltration, nanofiltration, reverse osmosis, and electrodialysis. This research explores the idea of creating potable water using a composite membrane containing date palm leaves. Polysulfone (PSF) was used as the base material, N-Methyl-2-Pyrrolidone (NMP) as a solvent, Polyvinylpyrrolidone (PVP) as the pore former, and date palm leaves powder as an additive. Water flux and contaminant removal are affected by membrane pore size. Dope solutions were prepared with five concentrations with 0.05%, 0.1%, 0.15%, 0.2%, and 0.25% additive powder weight. These portions were calculated as a percentage of the entire dope solution. A membrane's water flux test was conducted at 2bar pressure and room temperature. It was found that membrane containing 0.15% wt of an additive powder had the best water flux. Approximately 718.52 Litre per square meter hour (LMH) of water could pass through this membrane.

Introduction

Desalination and filtration of water can be improved to reduce water crises. Due to its low energy consumption, membrane technology is becoming increasingly popular. Additionally, membranes are made from natural materials that are generally simple to manufacture[1]. Several researchers have used nanomaterials such as carbon nanotubes and metal oxides. Materials like these are expensive. It is necessary to discover alternative natural materials that reduce the final filtration system's price. The main aim of this research is to test membranes for water permeability, conductivity, Total Dissolved Solids (TDS), pH, and contact angle using natural materials as additives.

To remove the dissolved solids from the water two stage filtration process was used. The Ultrafiltration (UF) process was followed by the Reverse Osmosis (RO). This study examined on a field scale the feasibility of polishing secondary effluents for unrestricted reuse, particularly for agricultural irrigation. Two parameters observed were plant height and stem diameter. Using RO effluents yielded the best plant development, and using secondary effluents from the reservoir yielded the poorest. Based on the findings of this study, irrigation with RO effluents with fertilizers provided the best plant development and ultimately the best yield for the poorest farmers [2].

Forward osmosis (FO) and pressure retarded osmosis (PRO) have become increasingly popular osmotically driven membrane processes (ODMPs). The process utilizes the difference in osmotic pressure between two water bodies to feed. They facilitate resource recovery from wastewater by treating water as well as generating electricity. In addition to being able to treat wastewater and

generate osmotic power, FO and PRO have shown tremendous ability in many filtration applications. Detailed and comprehensive studies on a large scale are required for further insights and evaluations of these processes [3].
The quality assessment of membrane bioreactor (MBR) treated wastewater was checked for its reuse in agricultural activities. It was noted that the utilization of membrane bioreactor (MBR) in conjunction with membrane filtration offers a more favorable alternative for the biological treatment process compared to the Conventional Activated Sludge Process (CAS), primarily due to its reduced spatial requirements. Membrane Bioreactor (MBR) was used as a pre treatment process before RO, thus giving improved results. The main aim of this particular research work was to identify, treat and assess MBR treated wastewater for its reuse in agricultural activities. For water quality analysis, water samples are classified into 3 classes. Various kinds of water counting tap water, MBR effluents, NF/RO permeates and the mixture of MBR/RO effluents (2:1) were analyzed and evaluated as irrigation water. The result indicated that MBR effluents with RO permeate at a volume ratio of 2:1 satisfied nearly all parameters [4].
The use of membrane technology in petroleum industry is for the process of production of nitrogen, sweetening of natural gas and removal of nitrogen, etc. for the largest number of applications RO membrane was utilized. Ultrafiltration process was used as a pre treatment for the RO feed. The study shows, even with the success of available membrane technology applications there are various challenges in front of membrane technology. Pre treatment steps and modification in material used for membrane preparation can help to reduce these challenges [5].
A nanomembrane or photocatalyst can be tailored to fit a specific application for a novel water technology. Membranes made from nanocomposite materials are capable of retaining particles and removing contaminants at the same time. As synthetic membranes become more and more complex and flexible, polymer brushes are increasingly appealing for surface modification. They also play a critical role in membrane separations. In order to keep membranes from fouling and scaling, polymer brushes were developed. In wastewater effluent streams, brush membranes were used to increase dye and small molecule rejection. Brush membranes are similar to commercial polyamide reverse osmosis membranes in terms of fluxes and salt rejections, but they are less likely to scale due to mineral precipitation [6].
In this research work the nanoparticles of silver were ingrained into the Polysulfonic membranes. This membrane was capable to remove the bacteria such as, Escherichia coli K12 and Pseudomonas mendocina KR1, and the MS2 bacteriophage. The hydrophilicity of a composite membrane was increased by incorporating silver nanoparticles. As per the X Ray Photoelectron spectroscopy analysis, the loss of silver particles from surface of membrane was observed for the short period of filtration. The surface of the membrane was found to contain 90% of silver particles by inductively coupled plasma mass spectrometry (ICP). Loss in silver particles indicates that there were some antimicrobial activities [7].
Carbon NanoTube (CNT) based membranes offer enhanced permeability and selectivity by combining the promising properties of carbon nanotubes with membrane separation technologies. For applications in water treatment, researchers examine the existing membrane architectures based on CNTs and their advantages and limitations. Several studies were conducted to provide insight into the potential of CNTs to overcome the limitations of conventional membrane technologies. Using computational simulations, Corry et al. concluded that the ideal CNT diameter to obtain good water permeability and salt rejection is <0.6 nm. Further computational studies have shown that adding chemical functionalities to the sidewalls of CNTs with large diameters also improves salt rejection efficiency. Membrane performance is most affected by the porosity of the membrane. The average size of the pore, the porosity, and distribution of pore size are the key factors affecting membrane separation efficiency. Water desalination and removal of contaminants can be achieved with CNT-based membranes [8].
Compared with polymeric membranes, ceramic membranes have a longer useful life expectancy, a higher permeate flux, a lower fouling propensity, a higher cleaning efficiency, and a lower environmental impact. On the basis of the life cycle of full-scale plants, several techno-economic

analyses compared ceramic membrane plants with polymeric membrane plants in terms of cost competition. A ceramic membrane can resist chemical oxidants and reactive species, which makes it ideal for use in new (photo) catalytic processes. As demand for ceramic membranes increases, their cost will further decrease. In order to improve product design and operate sustainably for long periods of time, further research is needed on different aspects, including inexpensive ceramic materials and fouling (biofouling) advancement [9].

In the process of water treatment, graphene membranes have become the key material due to rapid interest and research achievements. Researchers have briefly discussed adsorption, Donnan exclusion, and size exclusion within membranes. It has been observed that CNT-based membranes offer outstanding performance and characteristics in terms of both selectivity and characteristics, with potential applications from liquid to gas segregation. As well as polymeric membranes (Polysulfone, Polyacrylic Acid, activated polyacrylonitrile, and polyvinylidene fluoride), traditional membranes include metal oxide nanomaterials (TiO_2, FeO, Silica, Ag, and ZnO). Polymeric membranes, however, are limited in their industrial applications due to their pristine performance. Compared to pure polymeric materials, those altered with the addition of a nanoparticle-forming complex offer certain advantages, including lower fouling, higher selectivity, high diffusion rate, and permeability [10].

The immune system becomes more complex with age, and susceptibility to diseases is not solely determined by antibodies. This increases the likelihood of waterborne diseases among the elderly population. A study was conducted to examine the prevalence and predictors of waterborne diseases among the elderly in India. Data was collected from the Longitudinal Ageing Study in India (LASI), 2017-18. The study reveals that waterborne diseases are more prevalent among the elderly in rural areas (22.5%) compared to urban areas (12.2%) due to the use of unimproved water sources. Elderly individuals living in rural areas are more susceptible to waterborne diseases. The study also identifies variations in the prevalence of waterborne diseases among different states. [11].

The inclusion of carboxylated multiwalled carbon nanotubes (MWCNTs-COOH) into the PVDF/PVA UF membrane resulted in an enhancement of its hydrophilicity. This led to an increase in the pure water flux and an improvement in its ability to resist fouling. The composite membrane, which consisted of 0.09 wt% of MWCNTs-COOHs, exhibited the highest pure water flux, approximately double that of the PVDF/PVA membrane. Additionally, this composite membrane demonstrated an elevated bovine serum albumin flux recovery rate, showing a 17% increase. The porosity and dynamic contact angle of the composite membrane indicated a heightened level of membrane hydrophilicity. Furthermore, the modified membrane achieved a rejection rate of 91.0% for Dextran 600k. The mechanical properties of the membrane, such as break strength, elongation at break, and Young's modulus, also experienced improvement with the addition of MWCNTs-COOH [12].

A UF membrane made of polyvinylidene fluoride (PVDF) modified with polyvinyl alcohol (PVA)-SiO_2 was prepared using the Non-solvent induced phase separation (NIPS) technique. The inclusion of PVA-SiO_2 particles enhanced the hydrophilicity and anti-fouling properties of the PVDF membranes. In comparison to the pure PVDF membrane, the PVA-SiO_2 PVDF membranes exhibited higher pure water flux and better rejection of bovine serum albumin (BSA) [13].

The research centers its attention on the modification of materials, the modification through blending, as well as the modification of surfaces. The production of ultrafiltration membranes made from polyvinylidene fluoride (PVDF) involved the utilization of Polyvinyl alcohol (PVA) as an agent to modify the hydrophilic properties of the pore surface. The creation of PVDF UF membranes was achieved through the use of a coagulation bath containing PVA. The inclusion of PVA resulted in an enhancement of the membranes' ability to resist fouling [14].

The morphology of membranes obtained by precipitation can be explained by thermodynamic and kinetic factors. Phase diagram used to consider the role of thermodynamics. TiO_2 and PVP additives used in PVDF/DMAc solution and the demixing process and precipitation rate determined for PVDF/TiO_2/DMAc/Water and PVDF/PVP/DMAc/Water systems. The membranes' morphology predicted by comparing these parameters. Prepared membranes characterized by measuring permeability, porosity, and contact angle. Increasing additive concentration increased permeability and porosity until thermodynamic parameter exceeded relative kinetic parameter. PVP concentration of up to 5% resulted in a finger-like structure. TiO_2 concentration greater than 0.5% yielded a sponge-like structure [15].

Nanocomposite membranes for membrane bioreactors (MBRs) have been synthesized using surface-modified silver nanoparticles. Silver nanoparticles were immobilized inside the membrane matrix to enhance antimicrobial properties. The modified nanoparticles resulted in different morphologies and average pore sizes due to phase inversion changes. In addition to improving antifouling properties, surface-modified nanoparticles also have enhanced antimicrobial activity. As a result, MBR performance improved, including flux and COD removal. There was also an increase in flux recovery [16].

Negatively charged Polyethersulfone (PES)/Polyimide (PI) membranes were created to improve membrane performance. Surface properties like charge and roughness were analyzed to enhance Polyethyleneimine (PEI) nanofiltration layer assembly. The UF blend membranes had a highly charged surface, leading to over 95% rejection of BSA due to smaller pores, roughness, and electrostatic forces. The addition of PI reduced fouling. The NF membranes had high flux and improved salt rejection, with $MgSO_4$ having the highest rejection rate [17].

The impact of neat and modified nanodiamond nanoparticles on PVDF membranes was investigated. FTIR analysis showed successful immobilization of PVP onto ND nanoparticles. PVDF membranes were then embedded with different percentages of ND and P-ND nanoparticles. The study found that membrane hydrophilicity and water flux improved with increasing nanoparticle percentage. Additionally, the number of surface pores on PVDF/P-ND membranes increased. The performance of PVDF and PVDF/ND and PVDF/P-ND membranes was compared in an MBR system with pharmaceutical wastewater feed. The study concluded that PVDF/P-ND membranes had better antifouling properties compared to PVDF/ND membranes [18].

The present study has conducted an assessment of the utilization of direct membrane filtration and direct forward osmosis for the purpose of wastewater treatment. Both of these approaches have the advantage of being energetically favorable and compact in nature. In order to enhance the formation of particles prior to direct membrane filtration (DMF), the process of coagulation-flocculation can be employed. Moreover, the efficacy of direct forward osmosis (DFO) processes utilizing forward osmosis membranes was also evaluated. It is worth noting that the concepts of direct membrane filtration and direct forward osmosis demonstrate promise for the treatment of municipal wastewater, particularly for small and medium-sized waste water treatment plants (WWTPs) [19].

The utilization of the phase inversion technique was employed in the fabrication of the membrane. The experimental results of the ultrafiltration were carefully observed for the purpose of measuring the average pore size. It was observed that the blended membrane consisting of PEI/PVP exhibited the highest level of pure water flux, water content, and surface energy when 8 wt% of PVP was incorporated. However, the addition of PVP led to a reduction in tensile strength while simultaneously enhancing the resistance to fouling [20].

Researchers have created membranes using Al_2O_3 nanoparticles. They have investigated the effects of nanoparticle size and concentration on membrane properties and performance. A phase inversion method involving polyethylenimine and Al2O3 nanoparticles was used for membrane preparation. Various tests and measurements were conducted to analyze the membranes. It was observed that the presence of Al_2O_3 nanoparticles improves the permeability and selectivity of the membranes. The membrane containing 5 wt% 20 nm Al_2O_3 exhibited the best performance [21].

Two major research gaps identified are the limited materials being used for the synthesis of membrane and as an additive and the scarcity of suitable materials which can act as an additive for modification of membrane suitable for water purification.

Methodology

The membrane material should have good thermal, chemical and mechanical properties. The most likely used membrane materials are hydrophilic materials. The adsorption capacity of hydrophilic materials is less, hence the chances of membrane fouling reduces. There are few steps followed for the preparation of composite membrane. It includes preparation of fine powders of additive materials, formation of dopes followed by casting of membrane.

Date Palm Powder Preparation

Date Palm Leaf Powder: Date Palm leaves were collected and dried at 70ºC for a month, in the oven. The powder is made by the grinder followed by the sieving process. The fine powder was then separated using Muslin cloth having pore size between 50-100µm.

Fig. 1. Date Palm Leaves and Its Fine Powder

Dope Solution Preparation

a. N –methyl-2-pyrolidone (NMP) was used as a solvent for the dope solution. The amount added was 37.35ml i.e. the 75% weight of the entire dope solution
b. The amount of additives taken were 0.025gms, 0.05gmas, 0.075gmd, 0.1gms and 0.125gms of powders i.e. 0.05%wt, 0.1%wt, 0.15%wt, 0.2%wtand 0.25%wt respectively and added it to NMP solution.
c. The solution was kept in the sonicator for 15 minutes of time duration.
d. 3.6gms of polyvinylpyrrolidone and 9gms of polysulfone beads were added in the sonicated solution.
e. Screw capped bottles were kept in a shaker for 4 to 5 days until all the polymer beads get dissolved and a homogeneous solution was obtained.

Membrane Casting

The membranes were usually prepared from a polymer solution, which is a solution of two polymers (basic polymer and a pore former) in a suitable solvent, using a method known as "phase inversion" method. The synthesis of membrane involves pouring of above solution (referred to as dope) on the casting plate, allowing the plate to pass underneath a casting blade and then submerging the plate in water tank. The polymer solution precipitates in form of a thin polymer sheet [22].

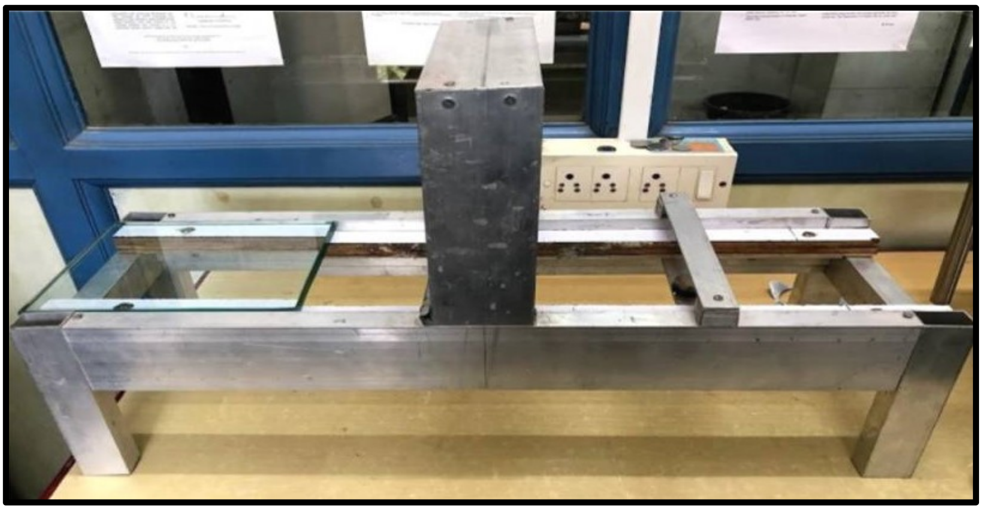

Fig. 2. Table Top Membrane Casting Machine

Membrane casting setup available in Pillai College of Engineering, New panvel, India was used for the preparation of membranes, manually. The setup consists of a railing, blade and the glass plate. To get the thin sheet of membrane following steps had been followed:

a. A small amount approximately 10ml was poured on the glass plate of the membrane casting machine
b. A glass plate was passed through a blade to obtain a thin rectangular membrane sheet
c. Then the plate was dipped in the contained filled with water
d. A rectangular shaped thin membrane was obtained

After membrane casting, membranes were kept in a container filled with water for 1-2 days so that solvent and pore former get completely dissolved in water due to which void spaces known as pores gets formed on the membrane.

Membrane Testing

This section has been divided into 5 parts. The Water Flux Test, Conductivity, Total Dissolved Solids (TDS), pH test of water samples before and after the filtration, Thickness and Hydrophilicity test.

Water Flux Test

The water flux of the above four types of membranes was measured using the UF Test Skid at Pillai College of Engineering, New Panvel. This equipment has been indigenously designed and fabricated [23]. Fig.3. is a photograph of the UF Test Skid. For the same purpose membranes were cut into the circular shape with the use of stainless steel stencil. The water flux is the main parameter considered to check the performance of the membrane. The UF Test Skid was used for testing the water flux of membrane.

Specifications of indigenously built Water Filtration Test Setup:

- 4 stainless steel test cells
- Test cells can be operated between 2 to 10 bar pressure
- Diameter of Test cell: 3.9 cm
- Active area of test cell: 11.94 cm^2

  The water not passing through the membrane is collected in reject tank which can be reused.

Fig. 3. Photograph of the UF Test Skid

Conductivity, TDS and pH Test

The conductivity of water samples before and after Ultrafiltration test was checked with the help of digital conductivity meter. After that the TDS value was calculated by using the following formula:
TDS= Correlation Factor* Conductivity
Where,
Correlation Factor is taken as 0.65 [24]
The pH value is measured with the help of digital pH meter.

Thickness Test

It is important to know the thickness of the developed membranes. Thickness testing of synthesized membranes was done by "Optical Interferometry and Microscope Extensometer." This facility is available at Pillai College of Engineering, New Panvel, India. Optical interferometry is the method used to measure the displacement by interference of light waves with high precision. This method is mainly used to measure distance in the nanometer scale. Extensometer is the device used for the tensile test, with the help of combination of these two techniques the thickness of membranes were obtained.

Contact Angle Testing

The contact angle testing was performed to get the information about the hydrophilicity of the membrane. Contact angle value is inversely proportional to the hydrophilicity of membrane, though it depends on the compositions and the elements present in the membrane [25]. The testing was done at Specialise Instruments Marketing Company (SIMC), Kanjurmarg, Mumbai. The theta optical tensiometer is used to find the value of contact angle.The software used is "OneAttension". Contact angles are measured for the membranes without an additive (Pure Psf), and 0.15%wt of date palm powder.

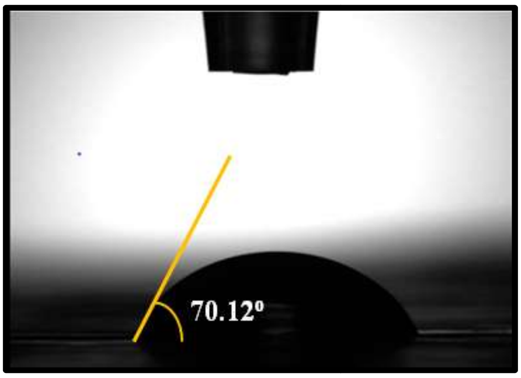

Fig. 4. Contact angle photograph for pure Psf

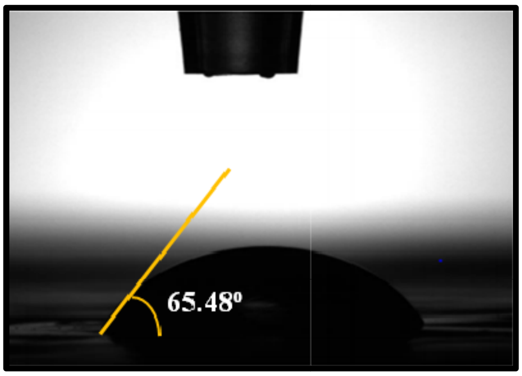

Fig. 5. Contact angle photograph for Date Palm

Observations

All synthesised membrane were then check for the water flux using Ultrafiltration Test Skid. Fig. 6 shows the water flux results of all the compositions made.

Observations for Water FluxTest

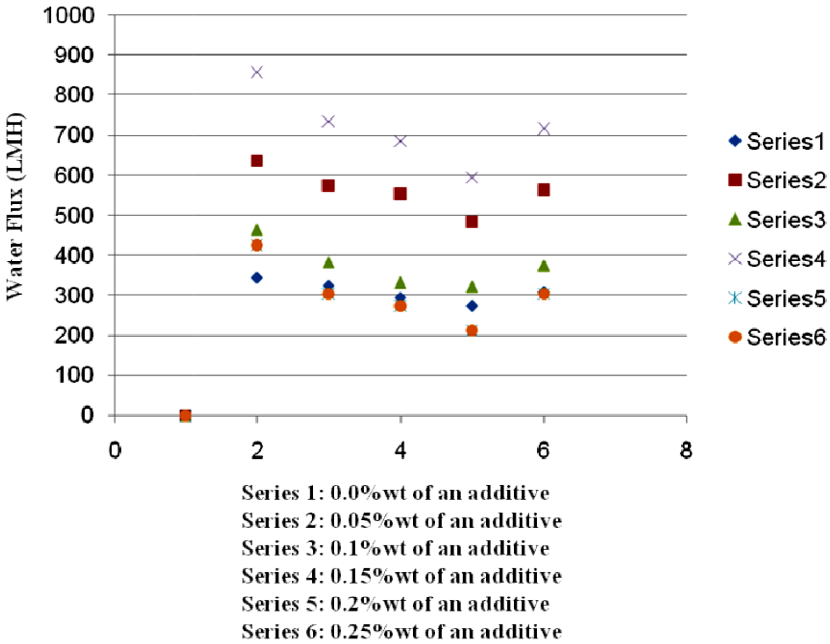

Fig. 6. Observation graph for Water Flux Test of Membranes (Without and with additive)

Date palm powder at 0.15% wt provides the highest water flux for the membrane. As the Contact Angle value of membranes with 0.15% wt of an additive is the least. The water flux of the membrane was reduced if the %wt of an additive material exceeds 0.15%, as it starts to clog the pores of membrane surface.

Calculation for Water Flux in Litres per Square Metre Hour (LMH)

The water flux is in LMH is calculated by the following formula:
Water Flux of membrane (F):

$$F = V/St. \tag{1}$$

Where,
F is Water Flux of Membrane ($L/m^2/h$)
V is Volume Flow Rate of Water (L)
S is Active Area of membrane (m^2)
t is Time (Hrs.)
F=718.52LMH

Observations for Conductivity, TDS, pH and Thickness:

- The conductivity of tap water is 144.5 MHOS/cm
- The TDS value for tap water is 96.81mg/L
- pH of tap water is 7.66

Table 1: Observation table for Conductivity, TDS, pH and Thickness

%wt of Date Palm Powder	Conductivity (MHOS/cm)	TDS (mg/L)	pH	Thickness (mm)
0.05	104.4	69.94	7.5	0.104
0.1	100.9	67.60	7.48	0.135
0.15	96	64.32	7.4	0.1027

Observations for contact angles of composite membrane

Table 2: Observation table for Contact Angle Measurements

Sample Name	Contact Angle (^{o})
Pure Psf	70.129
Date Palm Powder (0.15%wt)	65.48

Result and Conclusions

Result

Various characterization tests were performed to check the performance of synthesized membranes. Results for water flux, conductivity, TDS, pH and Thickness are given below.

Water Flux

Table 3: Results for Water Flux of Membranes

Membranes	Additives	Water Flux (LMH) measured at 2 bar for 5min.
Pure Psf	Nil	305.86
Psf + Additive (0.15%wt)	Date Palm Powder	718.52

Conductivity, TDS and pH

- According to the observations it was seen that, conductivity of membrane having 0.15%wt of date palm powder as an additive was least i.e. 96 MHOS/cm.
- It shows that the amount of Total Dissolved Solids in the water was also least i.e. 64.32mg/L.
- There was no promising change in the pH value of water sample after filtration.

Thickness

Thickness of membrane with 0.15%wt of date palm powder as an additive was 0.1027mm.

Contact Angle

The contact angle for the membrane with0.15%wt of date palm powder as an additive is less than the membrane of pure Psf.

Conclusions

Literature review indicates that various materials can be added to polysulfonic membranes to enhance their properties. There was a need to find an affordable and readily available material that could be used as an additive and show better properties. Water flux, conductivity, TDS, pH, and contact angle were improved with the membrane containing 0.15% wt date palm powder. The pure water flux of this membrane is almost twice of the pure polysulfonic membranes. There is a greater degree of hydrophilicity in these membranes. According to the experiments, the water flux of the membrane with Date Palm powder as an additive was 718.52LMH.

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