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Modification of PSU membrane by MWCNTs nanoparticle for Water Treatment Applications

A graduation project submitted to the Department of Physics in partial fulfillment of the requirements for the degree of Bachelor of Science in Applied Physics

by

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ABSTRACT

Water is an essential resource for life, and due to human activities, such as agricultural and industrial activities and energy production, the natural state of water has deteriorated, and this degradation is a concern as it increases the demand for water. In this context, it is important to consider alternatives to secure high-quality water.

The aim of this project is to modify Polysulfone membrane (PSF) by Multi-Walled Carbon Nanotubes (MWCNTs) nanoparticles for water treatment applications. To prepare the membrane, 17% Polysulfone polymer type 3500 was used. Two membranes were prepared, a pure Polysulfone membrane without any addition and a Polysulfone membrane with 1% MWCNTs. A number of characterization techniques and tests were performed. All testes were necessary to determine the effects of these parameters on the membrane, also the performance evaluation of the membrane efficiency in the desalination process using ultrafiltration, i.e. to obtain more flux and permeability, the carbon nanomembrane will be characterized by scanning electron microscopy (SEM), characterization with respect to the degree of contact angle, and the use of Thermogravimetric Analyzer (TGA), and tensile testing to measure thermal and mechanical properties.

The experimental results showed that the new addition of MWCNTs was effective in improving the membranes resulting in a better improvement in the water flow rate. This study also contributes to the development of membrane technology for water treatment applications.

الملخص

تعتبرالمياه مورداً أساسياً للحياة، وبسبب الأنشطة البشرية مثل الأنشطة الزراعية والصناعية وإنتاج الطاقة، تدهورت الحالة الطبيعية للمياه، يشكل هذا التدهور مصدر قلق لأنه يزيد من الطلب على المياه. وفي هذا السياق، من المهم النظر في بدائل لتأمين مياه عالية الجودة. **يهدف** هذا المشروع إلى تطوير غشاء البولي سلفون (PSF) بواسطة الجسيمات النانوية لأنابيب الكربون النانوية متعددة الطبقات (MWCNT)، لتطبيقات معالجة المياه. تم استخدام بوليمر البولي سلفون من النوع 3500 بنسبة 17% في تحضير الأغشية. تم تحضير غشائين,غشاء البولي سلفون النقي بدون أي إضافة ,وغشاء البولي سلفون المضاف اليه مادة MWCNTs بنسبة 1%. تم تحضير الأغشية ومن ثم تشخيصها وبعد ذلك اختبارها. كانت جميع الاختبارات ضرورية لتحديد تأثيرات هذه المعاملات على الغشاء، وكذلك تقييم أداء كفاءة الغشاء في عملية التحلية باستخدام الترشيح الفائق، و للحصول على مزيد من التدفق والنفاذية، تم تشخيص غشاء الكربون النانوي عن طريق المجهر الإلكتروني الماسح (SEM).)، والتشخيص بدرجة زاوية التلامس، واستخدام محلل قياس الحرارة الحراري (TGA)، واختبار الشد لقياس الخواص الحرارية والميكانيكية. أظهرت النتائج التجريبية أن الإضافة الجديدة لـ MWCNTs كانت فعالة في تحسين الأغشية مما أدى إلى تحسن أفضل في معدل تدفق المياه. تساهم هذه الدراسة أيضًا في تطوير تكنولوجيا الأغشية لتطبيقات معالجة المياه.

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شكراً للدكتورة /غادة خوقيرعلى كل ماقدمته لنا من علم ومعرفة وبذل الكثير من الوقت والجهد في سبيل تقديم الخبرة الكافيه لنا، شكراً للطاقم التعليمي بقسم الفيزياء على كل ماقدمتموه لنا من عطاء.

شكراً لمركز مدينة الملك عبدالعزيز للعلوم والتقنية لإتاحة الفرصة في إكمال مشروع التخرج لديهم , ونخص بالإشادة والشكر إلى الدكتور/مختار فال على توجيهاته ودعمه اللامحدود لنا , كما نشكرالمهندس/ تركي مانع بشكل خاص على توجيهاته وبذل الكثير من الجهد في مساعدته لنا في فهم الأمور الهندسية والتقنية المتعلقة بالبحث,حيث لم يوفر اي مجهود في سبيل زيادة خبرتنا العلمية.

شكراً لإثرائكم لنا بعلمكم, نسأل الله أن يُبارك لكم في عِلمكم ووقتكم وأن يفتح عليلكم بعطائكم أبواب الخير والفتوحات الطيبة.

أثير حسين العجمي & روان يوسف فلاته

ABBERVIATION

UF	Ultra-filtration
RO	Reverse osmosis
NF	Nanofiltration
NMP	N-Methyl-pyrrolidone
PSF	Polysulfone
SEM	Scanning electron microscope
EWC	Equilibrium Water Content
WHO	World Health Organization
TFC	Thin Film Composite
PES	Polyether Sulfone
PC	Polycarbonate
PVDF	Polyvinylidene Fluoride
PBI	polybenzimidazole
PA	polyamide
MWCNTs	Multi-Walled Carbon Nanotubes
CNTs	Carbon Nanotubes
PVP	Polyvinyl Pyrrolidone
TMC	Trimesoyl chloride
MPD	M-Phenylene Diamine
DIW	Deionized Water
TGA	The Thermogravimetric Analyzer
BSEs	Backscattered Electrons
MACH	Mechanical Properties
CA	Contact Angle

TABLE OF CONTENT

LIST OF TABLES

LIST OF FIGURES

CHAPTER 1

Theoretical background

Water is an essential resource for life that has been degraded by human activities such as agricultural, industrial, domestic, and leisure activities and energy production. In addition, population growth is a concern since it increases water demand. According to the World Health Organization (WHO), 50% of the population worldwide will be in countries characterized by conditions of water instability by 2025. In this context of increasing necessity and loss of water sources, it is important to consider alternatives to secure high-quality water [1].

To date, different new ways of water processing have been proposed to optimize the production and yield, considering also the reduction of production costs and time. Nowadays, one of these ways has been membrane technology, which was, for the first time, introduced by Bechold in 1907, who used ultrafiltration processes. Since that time, membrane-based technologies began to gain popularity as separation processes. They are among the most significant advances in chemical and biological process engineering. Membrane processes are well defined due to the membrane being a primary tool for separating different types of molecules [2].

Membranes are thin and flexible structures that play a crucial role in various biological and industrial processes. A membrane can be defined as a thin layer of material that acts as a barrier or separator between two different environments, controlling the passage of materials between them. Membranes can be natural, such as cellular membranes in living organisms, or synthetic, like filtration membranes used in water treatment processes.

1.1 Membrane

Membranes are made of a specialized filtration material; these fabricated materials serve as selective barriers in separation processes. There are different types of membranes, the most important type of these membranes is the TFC membrane, which consists of two main layers of these layers: an ultrathin selective top layer and a porous supporting bottom layer. These membranes are primarily used in water and wastewater treatment processes. Usually, these types are particularly used in RO and NF applications.

A key advantage of TFC membranes is that each layer can be independently controlled and optimized to achieve desired selectivity and permeability while maintaining process called interfacial polymerization, which creates an ultrathin selective layer through interfacial cross-linking between reactive monomers. This technology has dominated industrial applications due to its excellent combination of water flux and salt rejection capabilities [3].

1.1.1 Organic and Inorganic Membranes:

Organic (Polymeric) Membranes such as PSF and polyether sulfone (PES) membranes are commonly used due to their good chemical properties. Other types include cellulose acetate, polyimide, polyetherimide, polycarbonate (PC), polyvinylidene fluoride (PVDF), and polybenzimidazole (PBI).

Inorganic Membranes such as Ceramic membranes are made from metal oxides like alumina, perovskites, zeolites, and kaolin. Metallic membranes including stainless steel, nickel, and nickel-based alloys are also inorganic membranes. Carbon-based membranes are inorganic with excellent size-sieving properties.

In comparison between these two kinds, organic membranes are cheaper and more flexible but have lower stability, while inorganic membranes have better stability under harsh conditions like high pressure/temperature but are more expensive [4].

There are also different types of filtration processes, including Ultrafiltration (UF), Nanofiltration (NF), and Reverse Osmosis (RO) membranes. There are different structures of these types, such as spiral wound, hollow fiber, and flat sheet configurations.

The origin of membrane technology is in improving water quality and efficiency in various industrial processes. Additionally, it mentions the chemical and physical properties of these membranes, their operational parameters, and the significance of maintaining optimal conditions for effective filtration.

1.1.2 Types of membranes and their advantages and disadvantages:

Thin Film Composite (TFC) Membranes:

TFC membranes are specialized filtration materials consisting of an ultrathin selective barrier layer formed on top of a non-selective microporous substrate as shown in Fig 1.1. They are primarily manufactured through interfacial polymerization, where an ultrathin selective layer is created by interfacial cross-linking between reactive monomers on a porous supporting membrane. These membranes have achieved tremendous success in producing the right combination of flux and salt rejection, generating significant interest in industrial sectors. Advantages of TFC are excellent selectivity and water permeability, each layer can be independently optimized and superior mechanical strength. Their disadvantages are low chlorine tolerance susceptible to fouling.

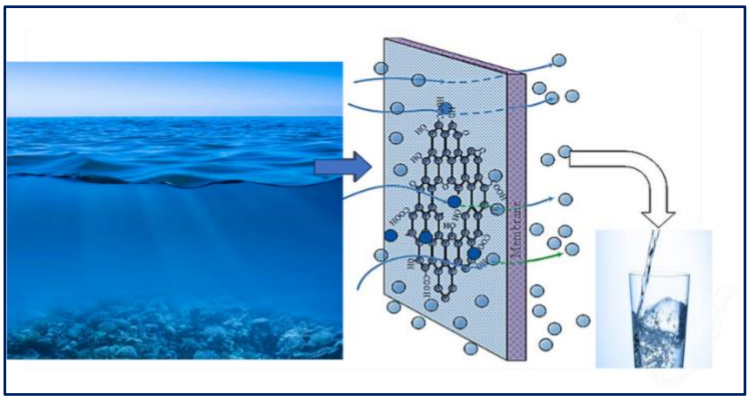

Fig 1.1: water filtration by membrane [11].

Asymmetric Membranes:

Asymmetric membranes are filtration materials manufactured through a single-step fabrication process. One of their key advantages is their ability to tolerate higher levels of feed water chlorine compared to composite membranes prepared from polyamide (PA). Advantages asymmetric membranes are better chlorine tolerance than TFC membranes, a single-step fabrication process.

Hollow Fiber Membranes:

Hollow fiber membranes are a specialized membrane configuration where forming a perfect polyamide film is particularly challenging. The membrane consists of hollow fibers where coating can be applied either on the outer surface or lumen (inner) surface. Advantages of Hollow Fiber Membranes are higher packing density than other configurations, and the disadvantages are difficult to form defect-free films and low reproducibility in commercial production. Research continues to focus on improving membrane properties, particularly in areas of fouling resistance, chlorine tolerance, and solvent stability [3].

1.2 Nanomaterials

Nanomaterials are materials with structural features at the nanoscale, typically between 1 to 100 nanometers in size. These materials exhibit unique physical, chemical, and biological properties that differ significantly from their bulk counterparts, particularly due to size-dependent characteristics. For example, nanomaterials may possess enhanced strength, chemical reactivity, or optical properties, allowing for applications in various fields such as medicine, electronics, and energy. They can be categorized based on dimensions into zero-dimensional (0D), one-dimensional (1D), two-dimensional (2D), and three-dimensional (3D) forms, as shown in Fig 1.2. Their exceptional properties stem from diverse structures, including nanoparticles, carbon nanotubes, and quantum dots, which can be synthesized through various top-down or bottom-up techniques to achieve specific functionalities.

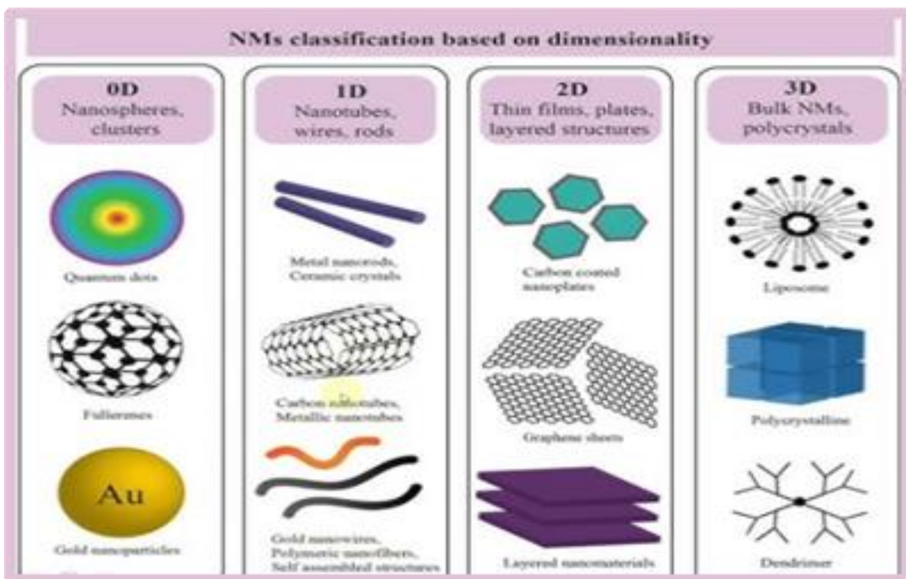

Fig 1.2: NMs classification based on dimensionality [12].

1.2.1 Properties of Nanomaterials

Nanomaterials exhibit unique physical and chemical properties that differ significantly from bulk materials. Their melting temperature decreases as particle size reduces, unlike bulk materials. The materials show enhanced mechanical properties including increased hardness, yield strength, and toughness compared to bulk forms.

Magnetically, non-magnetic bulk materials like gold and platinum can become magnetic at nanoscale. They demonstrate excellent chemical reactivity, stability, and catalytic properties including improved selectivity and reactivity [5]. These materials have significantly higher surface-to-volume ratios compared to bulk materials, leading to high surface energy and tendency to agglomerate. Their properties a (re largely determined by their shapes and sizes [5]

1.3 Nanofiltration (NF)

NF is a promising membrane-based separation technique for producing drinking water from different water sources. NF membranes typically have pore sizes between 0.5-2 nm and can effectively remove suspended solids, colloids, bacteria, and organics while partially removing dissolved ions as shown in Fig 1.3. Compared to traditional water treatment methods, NF offers great advantages including small footprints, ease of automation, wide-spectrum contaminant removal, and flexibility to adapt to different feed water qualities. It can achieve over 98% removal of calcium and magnesium ions through size exclusion. NF serves as an effective alternative to RO, requiring less energy while still providing good water quality. Technology has found widespread applications in surface water treatment, groundwater purification, and point-of-use water treatment systems. Recent advances focus on developing novel membrane materials and fabrication techniques to enhance performance while reducing energy consumption. Advantages of NF can be listed as follows, Effective Removal of Contaminants, Selective Permeability, Lower Energy Requirements, Broad Applicability, Compact Design and Ease of Operation, Control of Pathogens and Pollutants, Improved Water Quality, and Flexible Integration. NF membranes are increasingly used due to their ability to balance performance, cost, and energy efficiency, addressing diverse water quality challenges [6].

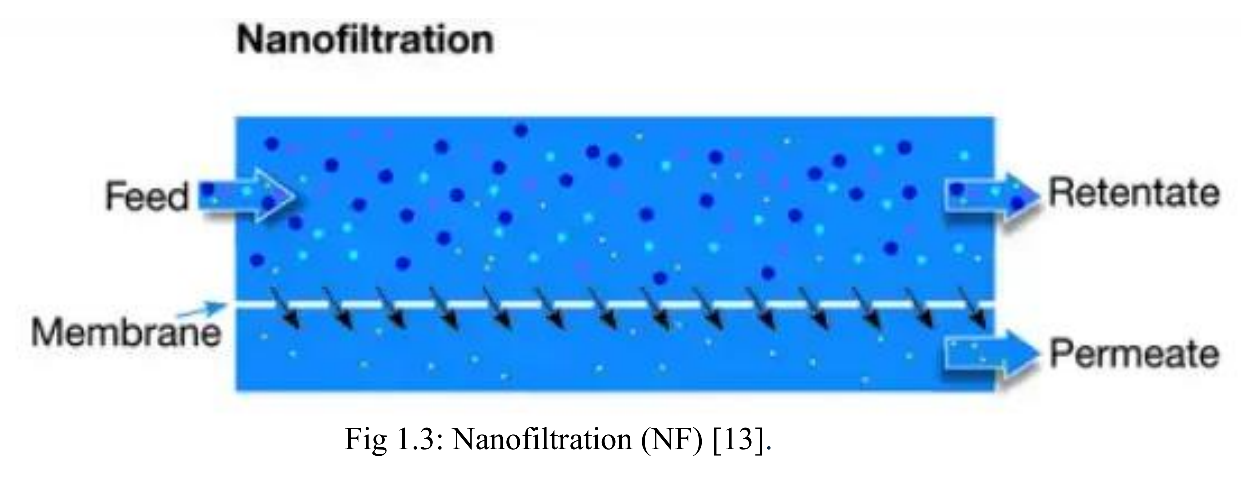

Fig 1.3: Nanofiltration (NF) [13].

1.4 Reverse Osmosis (RO)

Reverse osmosis is a process where water is forced through a semi-permeable membrane under pressure, separating contaminants such as salts, bacteria, and other impurities as illustrated in Fig 1.4. It is commonly used in desalination, water purification for drinking, and industrial applications.

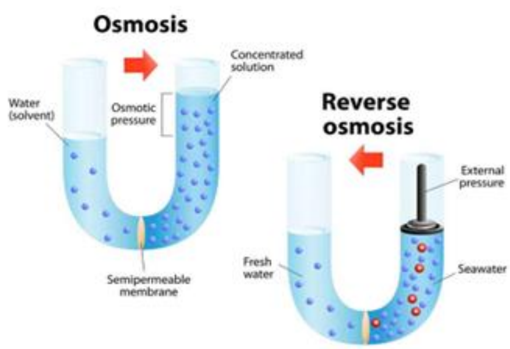

Fig 1.4: the fundamentals of reverse-osmosis [14].

1.5 Improving membranes

Optimization of the feed spacer geometry is one of the key challenges for improved ultrafiltration performance in water treatment and desalination.

i. Multi-Walled Carbon Nanotubes (MWCNTs).

Multi-walled carbon nanotubes (MWCNTs) can play a significant role in enhancing the properties of membrane capabilities through several important properties such as improving mechanical strength and stability, enhancing electron affinity, flexibility during modification/functionalization, and better permeability and selectivity

When incorporated into membranes, CNTs will enhance antifouling capabilities, prevent accumulation of inorganic salts and dissolved organics, improve removal of waterborne contaminants, and better desalination performance. These properties make CNT membranes particularly effective for water treatment and purification. Their unique morphological and physicochemical characteristics, combined with excellent water permeability and adsorption capacities, make them ideal for membrane separation processes. This has led to increasing research interest in CNT membrane development over the past decade [7].

ii. Polysulfone.

Polysulfone is a polymer material commonly used in membrane fabrication because it is inherently hydrophobic in nature, meaning it tends to repel or resist water due to its nonpolar hydrocarbon chains in its polymer structure. When used as a membrane material, polysulfone typically forms a structure with a dense top layer and porous sublayer. The surface properties of polysulfone membranes can be modified through surface treatment or by incorporating hydrophilic additives like polyvinyl pyrrolidone (PVP) to improve their performance. In membrane applications, polysulfone (typically 18% w/v) is dissolved in solvents like N-methyl pyrrolidone (NMP) to create the base membrane structure. This polymer serves as an important substrate material in desalination membrane fabrication as shown in Fig 1.5 [8].

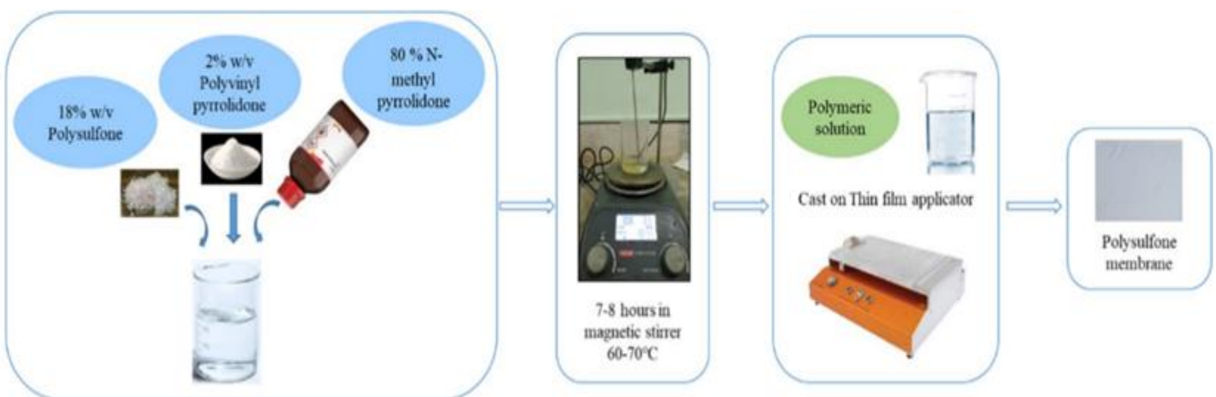

Fig 1.5: desalination membrane fabrication [8]

iii. Polyamide (PA).

Polyamide forms the active layer in thin film composite (TFC) membranes and has been widely used in desalination applications. It is created through an interfacial polymerization process that involves a reaction between m-phenylene diamine (MPD) and Trimesoyl chloride (TMC). The polyamide layer is formed on top of a support membrane through a cross-linking process, typically performed at 80°C for 10 minutes. When incorporated with additives like multi-walled carbon nanotubes (MWCNTs), the polyamide layer can demonstrate enhanced performance in terms of water flux and salt rejection as shown in Fig (1.6) [8].

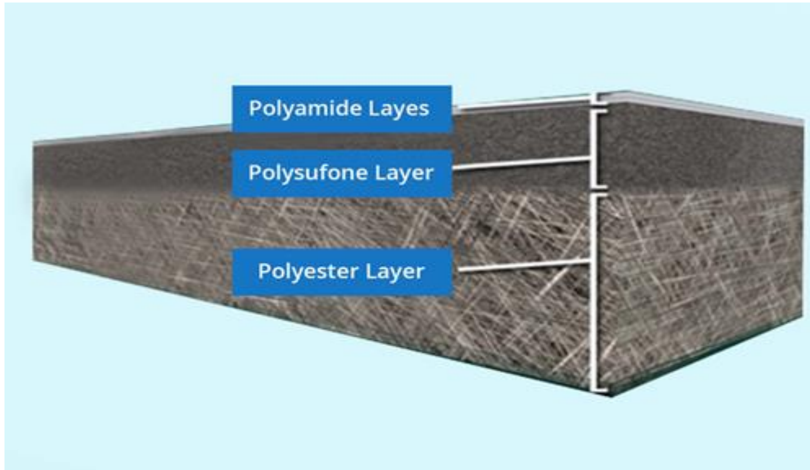

Fig 1.6: Image showing the layer of polyamaide [15]

1.6 Membrane restraints

Despite the development of membranes and attempts to improve their quality, there are still some obstacles that may reduce the efficiency of the membranes, such as:

Fouling Issues such as Biofouling require special attention for better membrane performance. Salt particle accumulation reduces membrane efficiency. Concentration polarization affects membrane performance.

Performance Limitations in achieving improved water permeability without compromising selectivity is challenging. Hydrophobic nature of certain membranes (like Polysulfone) can limit water flux. Pressure variations can affect rejection rates and membrane performance.

Structural Challenges in membrane thickness uniformity issues during casting. Surface wrinkles can form during fabrication. Pore size control and distribution challenges.

Operational Issues in performance degradation after regeneration. Water flux reduction over time. Salt rejection efficiency decreases with increased pressure beyond certain limits [8].

1.7 Literature Review

H. Guo *et.al.,* 2022 studied the TFC membranes, especially those made of aromatic polyamide, as reverse osmosis applications. Researchers have explored various surface modification approaches to improve fouling resistance. One promising modification involves coating membranes with thermo-responsive polymers such as P(NIPAM-co-Am), which has shown significant improvements in: water permeability (12% increase), salt rejection (98.5% to 98.9%), and fouling resistance (35% vs. 54.6% reduction in flux with BSA polymers) [6].

Ahmad *in* 2022, examined fabrication parameters' effects on membrane morphology and performance 46, while advances in membrane materials include novel two-dimensional and three-dimensional structures such as graphene, carbon nanotubes, and metal-organic frameworks. including thin-film composite membranes showing high water permeability and salt rejection, and sustainable approaches using renewable materials. Research has also focused on improving chemical, mechanical, and thermal properties for industrial applications. Additionally, membrane modification strategies like interfacial polymerization and dip-coating [4].

Abera and Mekuye *in* 2023, synthesized nanomaterials with physical, chemical, and electrical properties that remain size independent. These materials can be synthesized through two main methods: Top-down methods (including lithography, mechanical milling and laser ablation) and bottom-up methods (chemical and biological synthesis). Applications include fields as diverse as agriculture and electrical engineering. Specifically, nanomaterials are used in solar cells and OLEDs. Green synthesis methods using microorganisms and plant materials have emerged as an environmentally friendly approach [5].

Megha in 2024, conducted a research study whose main objectives were to synthesize an efficient desalination membrane with high salt rejection and water flux capabilities. The researchers aimed to improve the performance of the membrane by incorporating polyvinylpyrrolidone and multi-walled carbon nanotubes into the polymeric matrix. The key results showed that membranes with 0.01% MWCNTs achieved impressive performance, demonstrating: 92% salt removal rate, Water flux of 41 L/m^{2}h, better performance compared to standard PA membranes [8].

Rosen in 2014, designed an antimicrobial membrane using polymer colloids, multi-walled carbon nanotubes and silver nanoparticles. The results showed good antimicrobial activity against S. aureus and E. coli, especially with silver nanoparticles generated ex situ. The water flux was higher than the ethanol flux in the composite membranes, indicating increased hydrophilicity of the active layer. The test showed minimal variation in flux over time, indicating good stability. The membrane design was validated by multiple characterization techniques including SEM, FT-IR spectroscopy, and antimicrobial testing [9].

In 2011, Velu s *et.al.* studied the effect of two solvents (DMF and DMAc) on Polysulfone (PSf) and polyurethane (PU) ultrafiltration membranes. The performance results were that membranes containing 20/80 wt% PSF/PU using DMAc had a higher water flux (60.5 L/h) compared to DMF (23.5 L/h). Increased water content with higher polyurethane content in DMAc membranes yielded better results than DMF membranes. Protein permeability studies showed higher flow rates for membranes containing DMAc [10].

CHAPTER 2
METHODOLOGY

2.1 Preparation of Membranes

Two kinds of membranes were prepared using Polysulfone (PSF) type 3500. The first one is prepared of the polysulfone membrane without any additives. The second one is prepared of the polysulfone membrane with addition of 1% (MWCNTs).

Table 2.1: Composition of various membrane casting solutions.

Membrane	PSF-3500 (wt. %)	NMP (wt. %)	Additions (wt. %)
M_1	17	83	___
M_2	17	82	1

2.1.1 Pure Polysulfone membrane

First, 18% Polysolphon (PSF) was left to dissolve in a solution of 82% (NMP) Dimethylacetamide and 99% (NMP) for 24 hours at room temperature using a magnetic motor after which it is placed in a vacuum desiccator for an hour to remove the gas and air molecules from the serum after that we start the casting process The membranes were prepared through the phase inversion method. The casting solution was poured onto a clean and dry glass plate and cast at room temperature using a casting knife (gap height of 200 µm) with a constant casting speed of 40 mm/s by employing an automatic thin-film applicator After that, the glass plate was immersed into a coagulation bath filled with distilled water at room temperature. The membrane film was left to detach from the glass plate. Finally, the membrane was rinsed and stored in a plastic container containing distilled water**.** Fig 2.1 and Fig 2.2 shows the composition of the fabricated membranes.

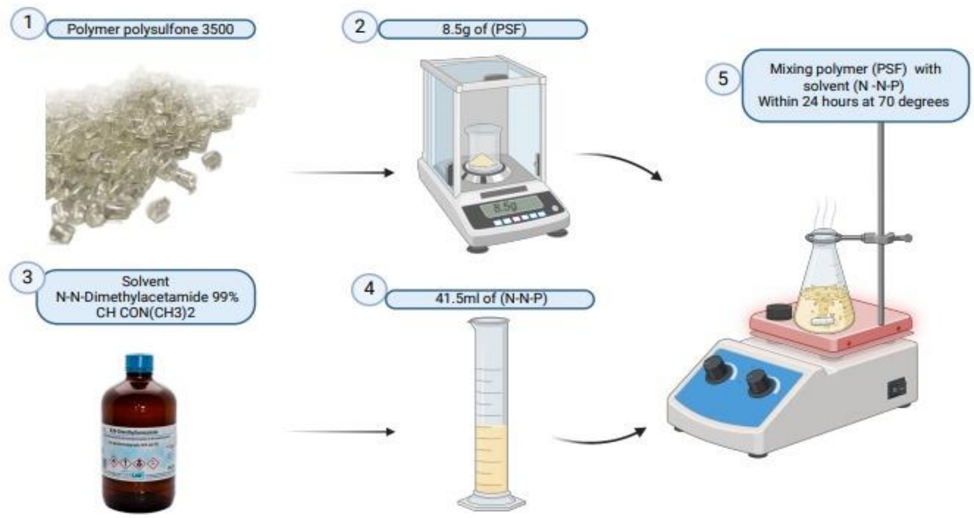

Fig 2.1: The process of preparing the Polysulfone membrane without any additives.

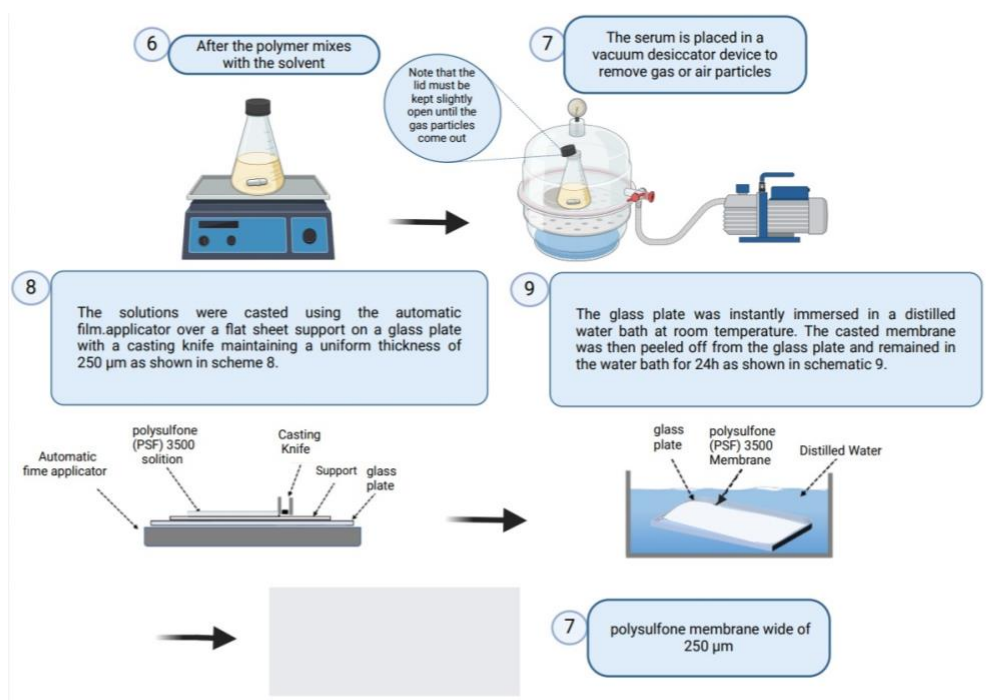

Fig 2.2: The process of preparing the Polysulfone membrane without any additives.

2.1.2 Polysulfone membrane with addition of 1% (MWCNTs).

In this stage, Nanomaterials were added to improve porosity and hence to enhance the permeating flux. Following the same steps as Stage 1, Polysulfone (PSF) type 3500 was used at a concentration of 17%. Which is equivalent to 0.17 grams, and the solvent (NMP) was used at a concentration of 82%, which is equivalent to 41 ml. 1% of (MWCNTs) was added in the form of powder. MWCNTs were initially dispersed in (NMP) solvent solution of 41 ml. Under ultrasonic treatment, to ensure diffusion and to ensure that no clumps remained in the solution. After the components were fully dispersed, the Polysulfone was added to the solution using a funnel. The mixture was then left to blend at a temperature of 70°C for 24 hours. As illustrated in Fig 2.3, After the dissolution period, the solution was placed under pressure for one hour to remove any possible air bubbles. Then, the same steps from Stage 1 were repeated to prepare the membrane. As illustrated in Fig 2.4.

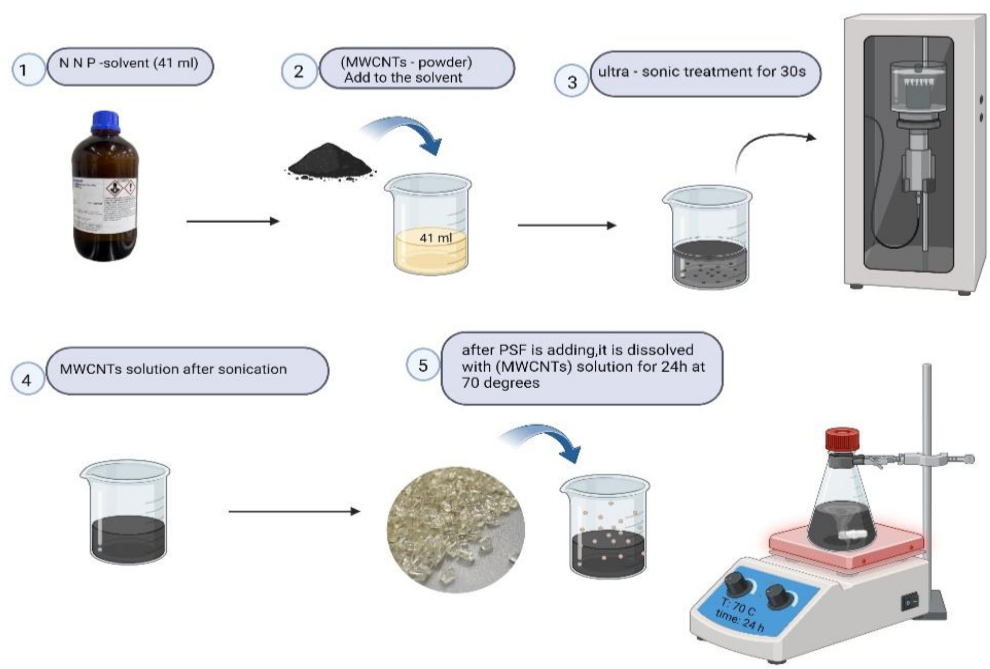

Fig 2.3: The process of preparing the Polysulfone membrane with the additive of carbon nanotubes.

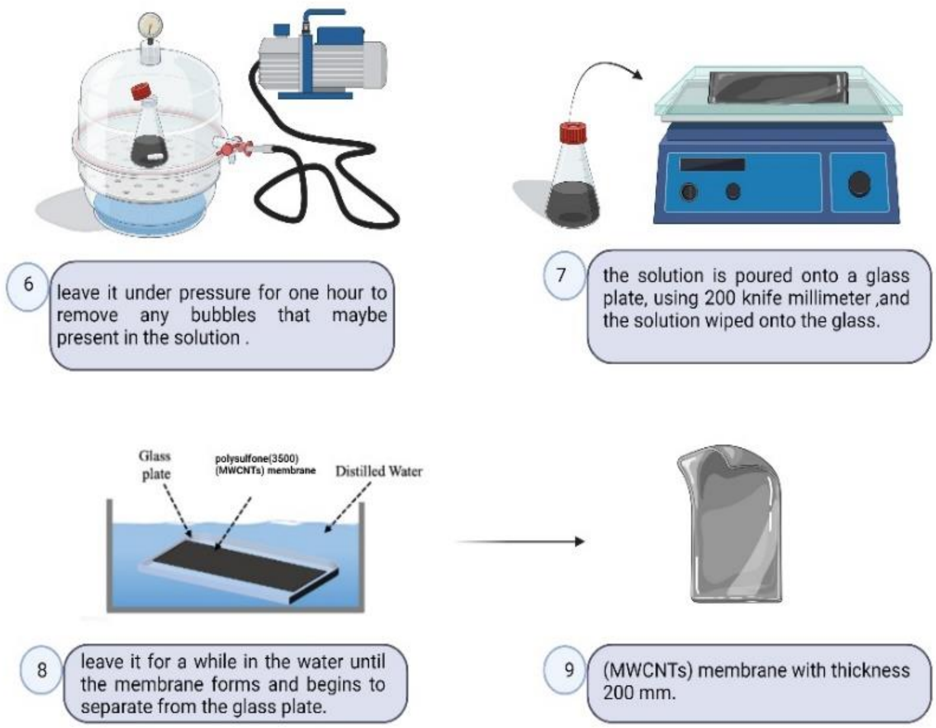

Fig 2.4: The process of preparing the Polysulfone membrane with the additive of carbon nanotubes.

2.2 Performing tests on the membrane

2.2.1 Contact Angle

The device is used to measure the contact angle between a liquid and a solid surface. This angle depends on the interaction between the liquid and the surface, serving as an indicator of the adhesive strength between them Fig 2.5.

Device function: Determining the wettability of the surface: The contact angle reflects the surface's ability to absorb the liquid. If the angle measures small on the outside of the drop, the surface is highly wettable (the surface is hydrophilic) Fig 2.6.a. On the other hand, a large angle indicates that the surface is non-wettable (hydrophobic) Fig 2.6.b.

Fig 2.5: contact angle device.

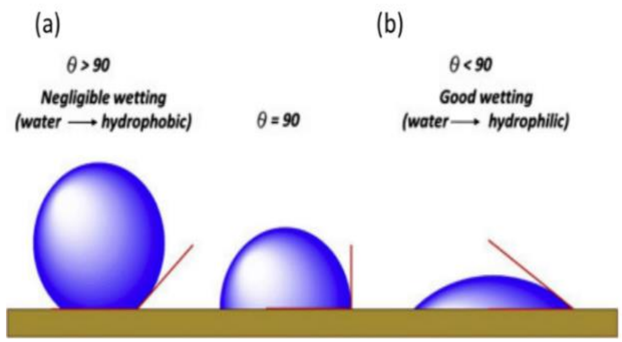

Fig 2.6: Image of hydrophobic and hydrophilic surface angles.

2.2.1 Contact angle teat method

The water resistance of a membrane can be physically assessed by measuring the contact angle of the membranes. Contact angle measurements were performed using an optical tensiometer. In this method, the membrane samples were first dried by placing them in an oven for several hours until the samples were completely dry of water and then we fixed the sample on a support, after which a drop of distilled water was deposited on a piece of the membrane Using a micropipette, after that we calculate the contact angle of the membrane with the drop of distilled water and determine the quality of the membrane, if the membrane is hydrophilic, the external contact angle will be small, but if the membrane is hydrophobic, the external contact angle will definitely be large. Fig 2.7

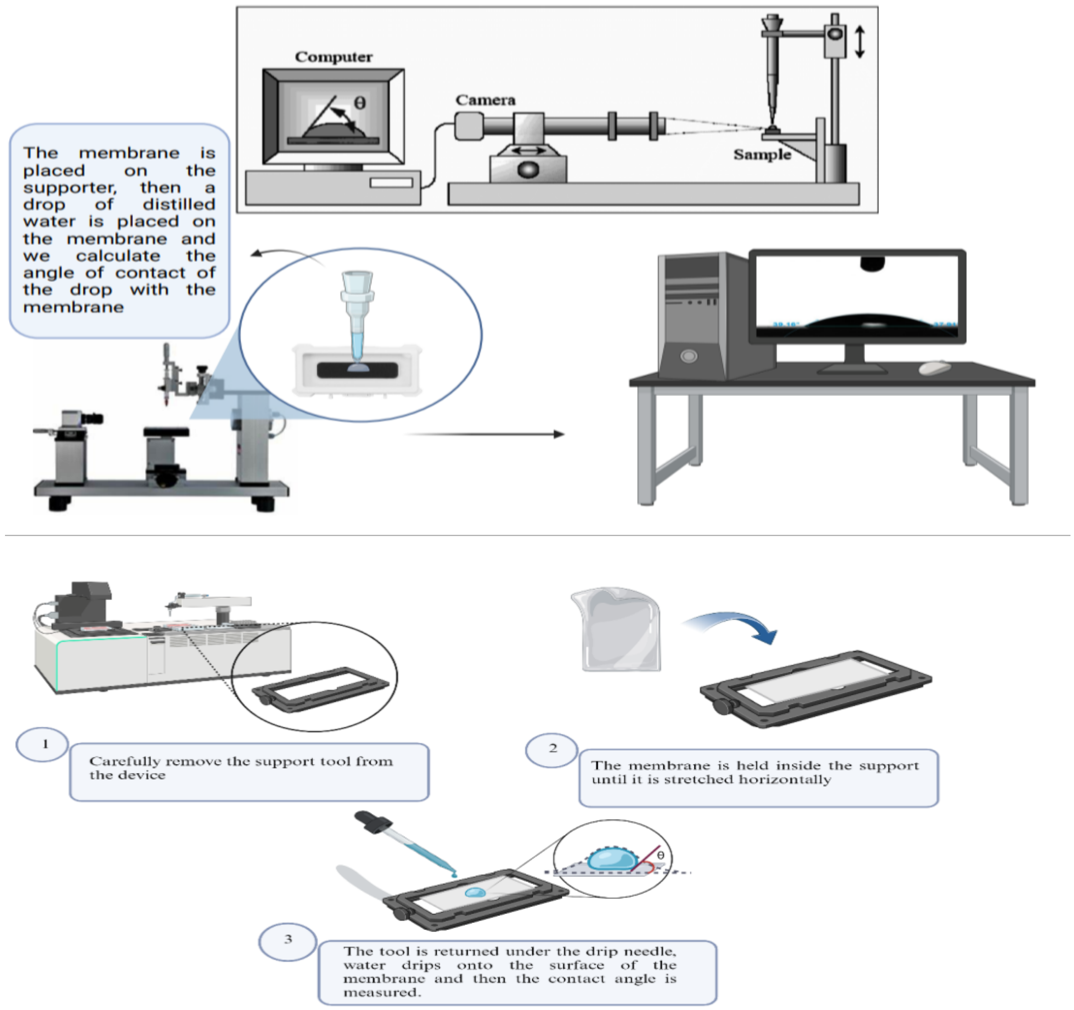

Figure 2.7: contact angle test method

2.2.2 The Thermogravimetric Analyzer (TGA)

The device is used to analyze the thermal properties of materials by measuring the change in mass as a function of temperature Fig 2.8.The TGA device operates by gradually heating the sample

Fig 2.8: TGA Device.

while measuring the change in its mass. Mass loss may occur due to evaporation, thermal decomposition, or combustion, and these changes are recorded as a function of temperature. Based on this data, material properties such as decomposition temperature, moisture content, and the presence of volatile substances can be inferred.

2.2.2 TGA test method

The aim of this test is to know the tolerance or resistance of the membrane to high temperatures by interpreting the results physically through the thermogravimetric decomposition rate graph. First, the membrane is dried in a dryer for an hour or more until the water in the membrane is completely evaporated. Before placing the sample inside the device, a material of aluminum powder was placed inside the crucible designated for the sample to ensure that the sample does not stick to the crucible after burning. After re-zeroing the scale, the sample is placed on top of the powder, and the weight of the sample should range from 10 mg to 30 mg Fig 2.9. The device was programmed to work in a system divided into five steps, as follows:

1) Heat from 30.00 C to 50.00 C at 10.00 C/min

2) Hold for 10.0 min at 50.00 C

Switch the gas to Oxygen at 20.0 ml/min, When the segment starts

3) Heat from 50.00 C to 500.00 C at 10.00C /min

4) Hold for 10.0 min at 500.00 C

5) Heat from 500.00 C to 1000.00 C at 15.00 C/min

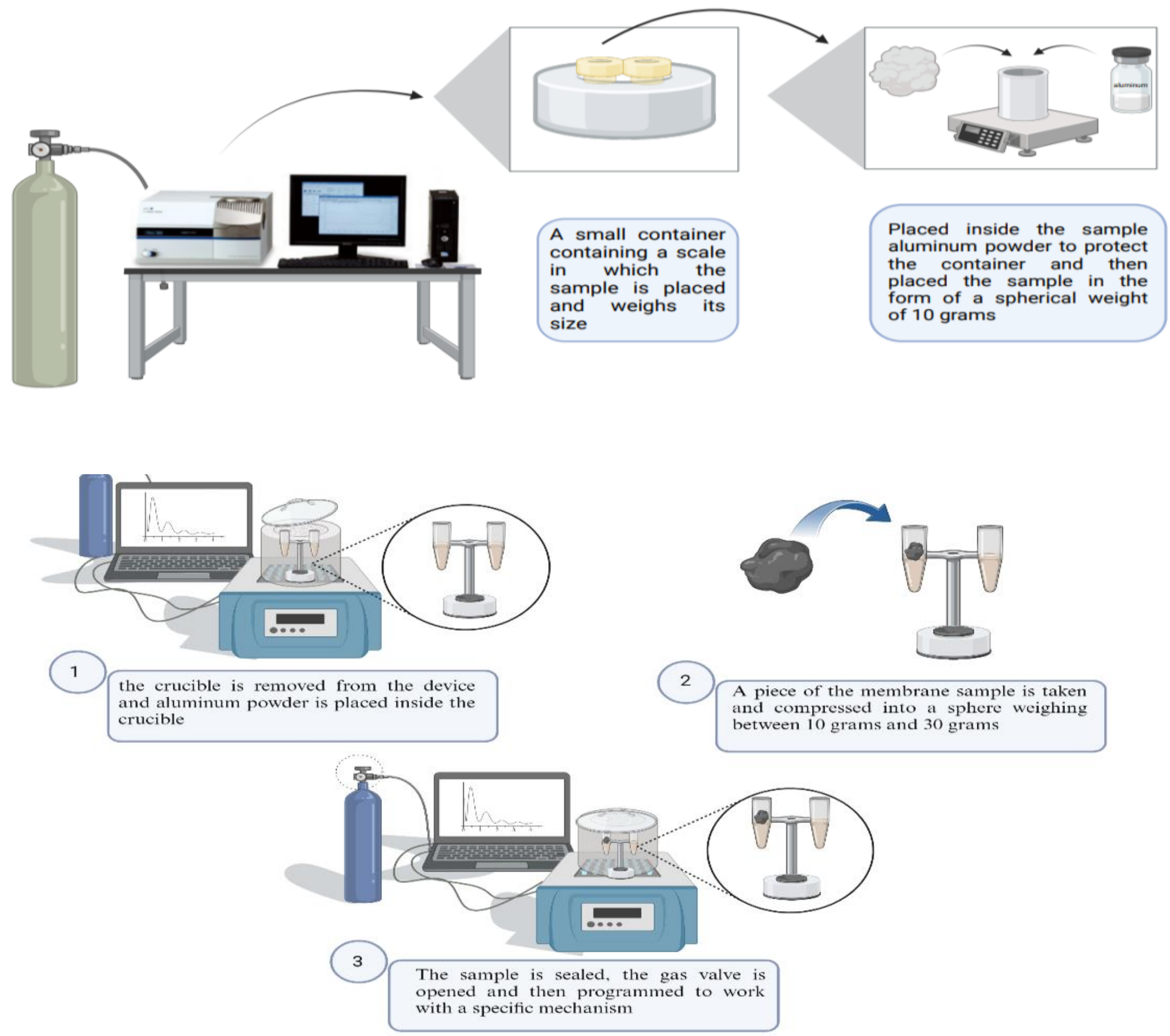

Figure 2.9: TGA test method

Switch the gas to Nitrogen at 20.0 ml/min, When the segment starts.

2.2.3 Scanning Electron Microscopy (SEM)

The Scanning Electron Microscope (SEM) stands out as a powerful imaging tool that provides exceptional depth of field and high-resolution examination of surface topography, see Fig 2.10. One of its greatest features is its remarkable depth of field capacity, allowing large objects to be imaged entirely in focus.

The device requires careful consideration of voltage settings - lower voltages (around 2-5 kV) work well for surface detail, while higher voltages may cause deeper beam penetration. For biological specimens, the conventional SEM requires water removal, and specimens must be properly preserved and mounted to reduce artifacts.

The SEM's effectiveness depends on balancing factors like working distance, voltage, and proper specimen preparation to achieve optimal imaging results.

Fig. 2.10: Scanning Electron Microscopy (SEM).

The device generates two types of signals: backscattered electrons (BSEs) from elastic scattering, and secondary electrons from inelastic events. Biological specimens typically require special consideration

For the sample preparation, dried membrane samples of an appropriate size to fit in the specimen chamber were mounted rigidly on a specimen holder called a specimen stub. Samples, approximately 1 cm2, were secured to the specimen stub by a carbon-based, double sided sticky tape called a tab holds the specimen to the stub, allowing an electrically conductive connection between substrate and sample surface. Prior to the SEM analysis, stubs were placed in vacuum chamber for platinum sputtering, this makes membrane surface electrically conductive and makes the instrument capable of operation in a low vacuum mode. Morphology studies were conducted at an acceleration voltage of 5 and 10 kV and a working distance of 8 mm followed by varying magnifications. For the cross-section analysis, the samples were oriented perpendicular to the incoming light/electron beam.

2.2.3 SEM test method

SEM is a method for high resolution surface imaging. The SEM uses electrons for imaging, much as light microscopy uses visible light, see Fig 2.11. The advantages of SEM over light microscopy include greater magnification (up to 100,000X) and much greater depth of field.

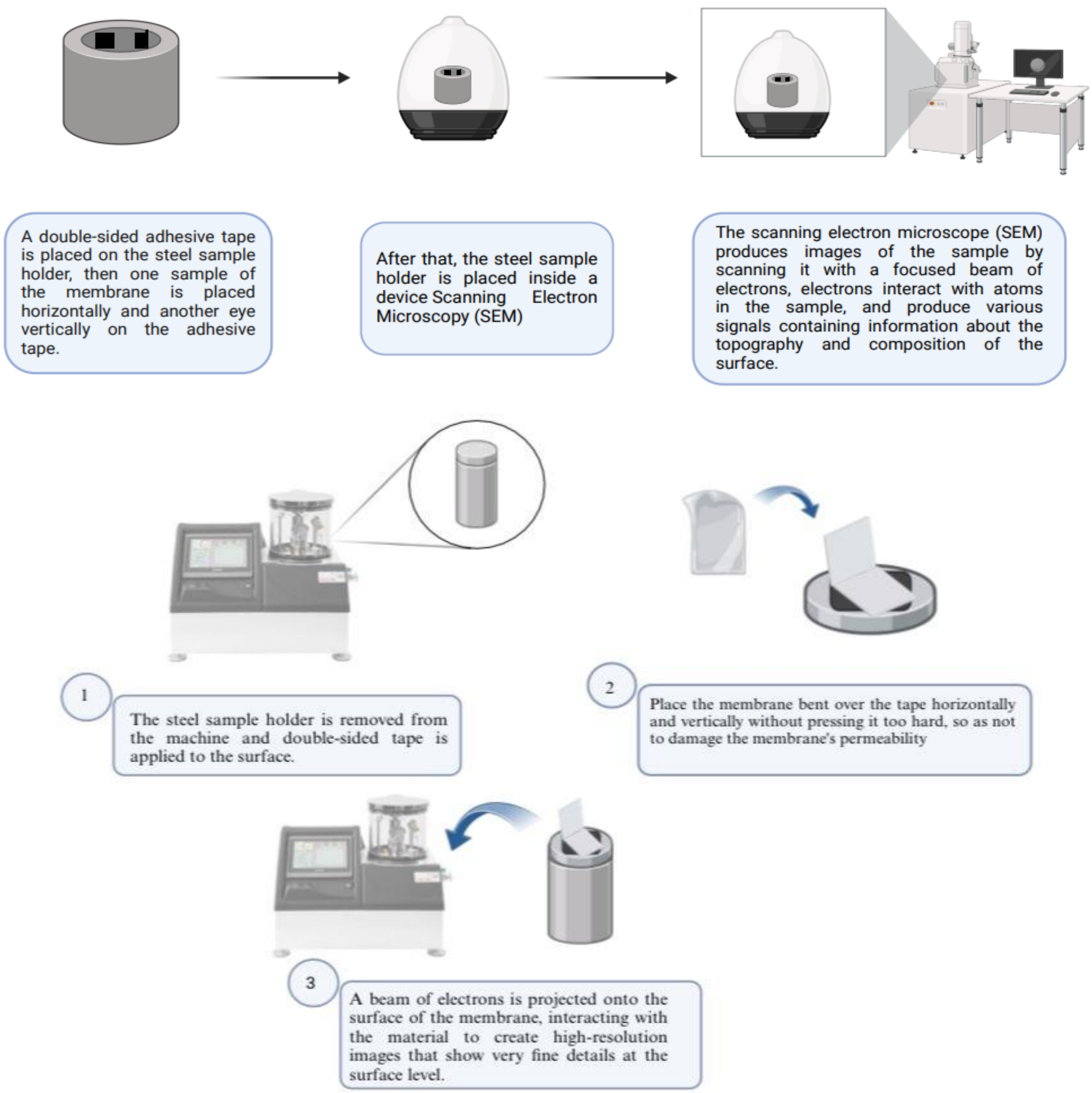

Figure 2.11: SEM test method

2.2.4 Mechanical properties.

A tensile test, also known as a tension test, is a strength test in which a sample experience controls tension until failure. Test results are commonly used to select a material for an application, quality control, and predict how the material will react under other types of forces. The mechanical strength of the membranes was checked by testing the tensile strength. Fig 2.12.

Fig.2.12: Mechanical properties Measurement Device.

CHAPTER 3
RESULTS

In this chapter, the preparation of MWCNTs nanomembranes for the application of ultrafiltration was investigated in this work. A number of characterization techniques and tests were performed, these experiments were necessary to determine the effects of these parameters on the membrane, also the performance evaluation of the membrane efficiency in the desalination process using ultrafiltration, i.e. to obtain more flux and permeability, the carbon nanomembrane will be characterized by SEM, characterization with respect to the degree of contact, and the use of TGA and tensile testing to measure thermal and mechanical properties.

3.1 Equilibrium Water Content (EWC)

The results shown in the following table show the extent of water saturation inside the membrane. We measured the membrane while it was wet, then dried it completely and weighed it again to compare the difference between the two weights and confirm the presence of pores inside the membrane, as shown in Table 3.1.

Table 3.1: equilibrium water content

Membrane (wt.)	W_W	W_D	Saturation (%)
M_1	**0.026**	**0.07**	**73.05**
M_CNTs	**0.050**	**0.010**	**80**

3.2 Contact Angle

By using a contact angle device, we measured the contact angle of the distilled water droplet on the surface of the membrane, and we found after the results that the angle of the first sample was smaller than the angle of the second sample so that the first angle was 38.9° for M_1, Fig 3.1.And the second angle was 60.1° for M_CNTs, Fig 3.2.Which shows the difference made by the addition of the optimizer, as shown in Table 3.2.

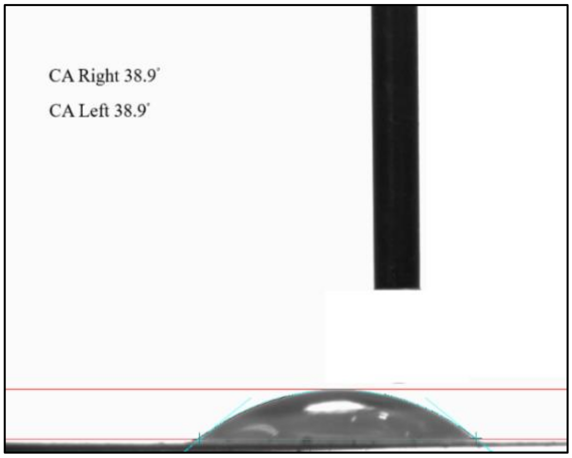

Fig 3.1: contact angle images for M_1

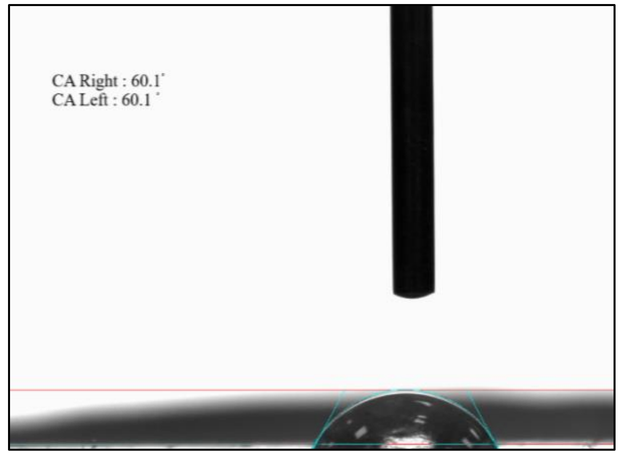

Fig 3.2: contact angle images for M_CNTs

Table 3.2: The results of contact angle

Membrane	Angle (°)
M_1	**38.9**
M_CNTs	**60.1**

3.3 Scanning Electron Microscopy (SEM)

SEM images of the cross-section of the membranes were taken as shown in the Figures From the cross-sectional images, it was observed that the pores of the membranes appeared in straight finger structures with open ends and a thin top skin layer. The effect of mixing MWCNTs nanoparticles in NMP solution can be appreciated, as a drastic change in morphology can be seen in the nanocomposite membranes (M_CNTs) when compared to the pure PSF membrane (M_1), as shown in Fig 3.3 and Fig 3.4.

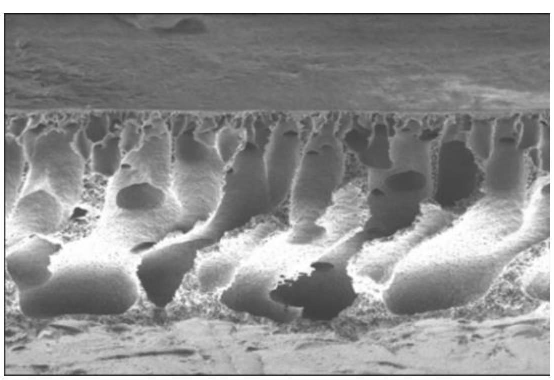

Fig 3.3: SEM images of the cross-section of the membranes for M_1

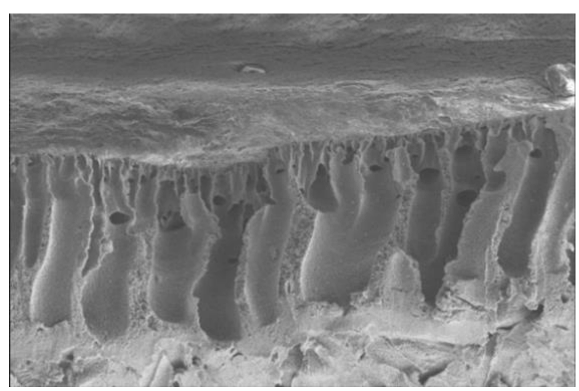

Fig 3.4: SEM images of the cross-section of the membranes for M_CNTs

3.4 The Thermogravimetric Analyzer (TGA)

The thermal properties of the nanocomposite membranes were investigated at temperatures ranging from 30 °C to 1000 °C. According to Fig 3.5 and Fig 3.6, the results showed that the addition of MWCNTs to the PSF membrane enhanced the thermal stability of the membrane in a very small and barely noticeable way. It can be seen from the results that the small increase in thermal stability of the nanocomposite membranes is due to the strong interfacial bonding between the PSF structure and the MWCNTs nano-additives.

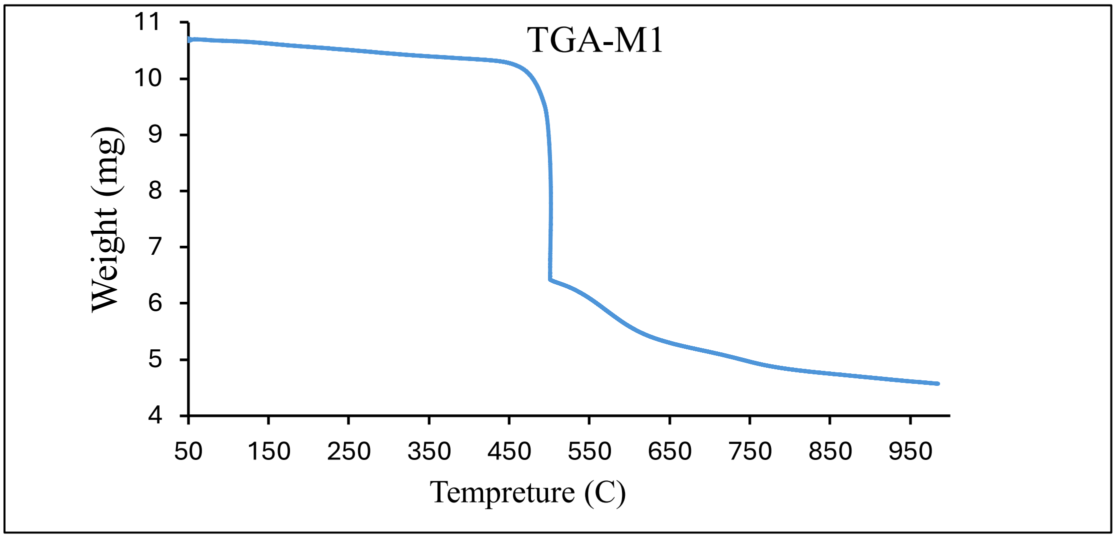

Fig 3.5: TGA curves of as a function of temperature for M_1

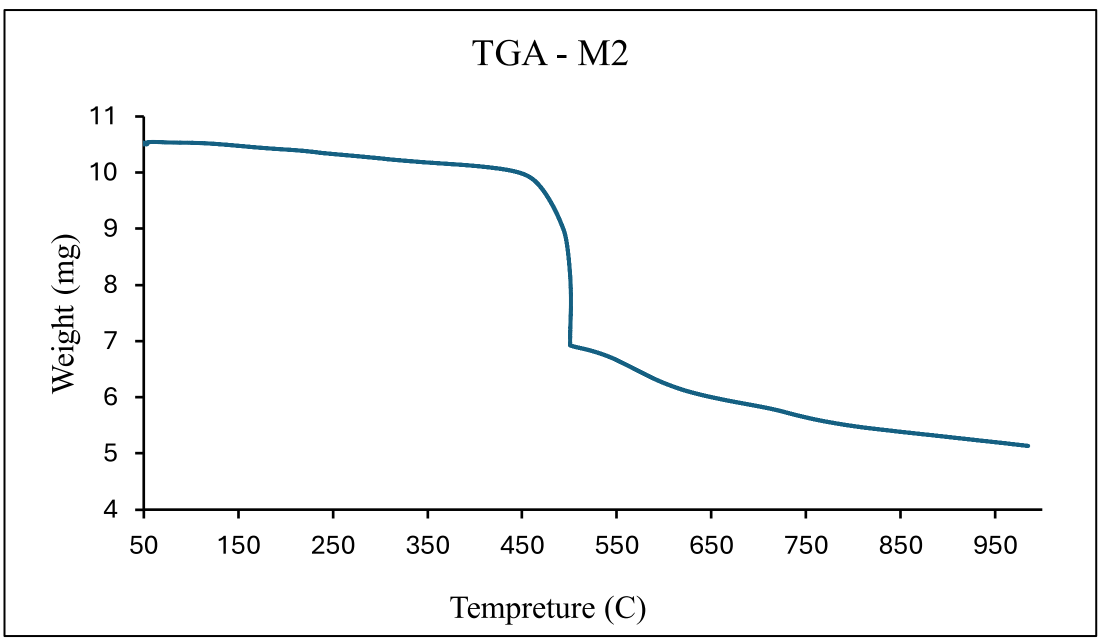

Fig 3.6: TGA curves of as a function of temperature for M_CNTs

3.5 Mechanical properties

Fig 3.7 shows the mechanical properties of the membranes. As shown in the curve of stress-strain measurements, the pure PSF membrane exhibited a tensile strength of 1.39 ± 0.1 MPa. This value increased to 1.49 ± 0.1 MPa when 1 wt% of MWCNTs were added.

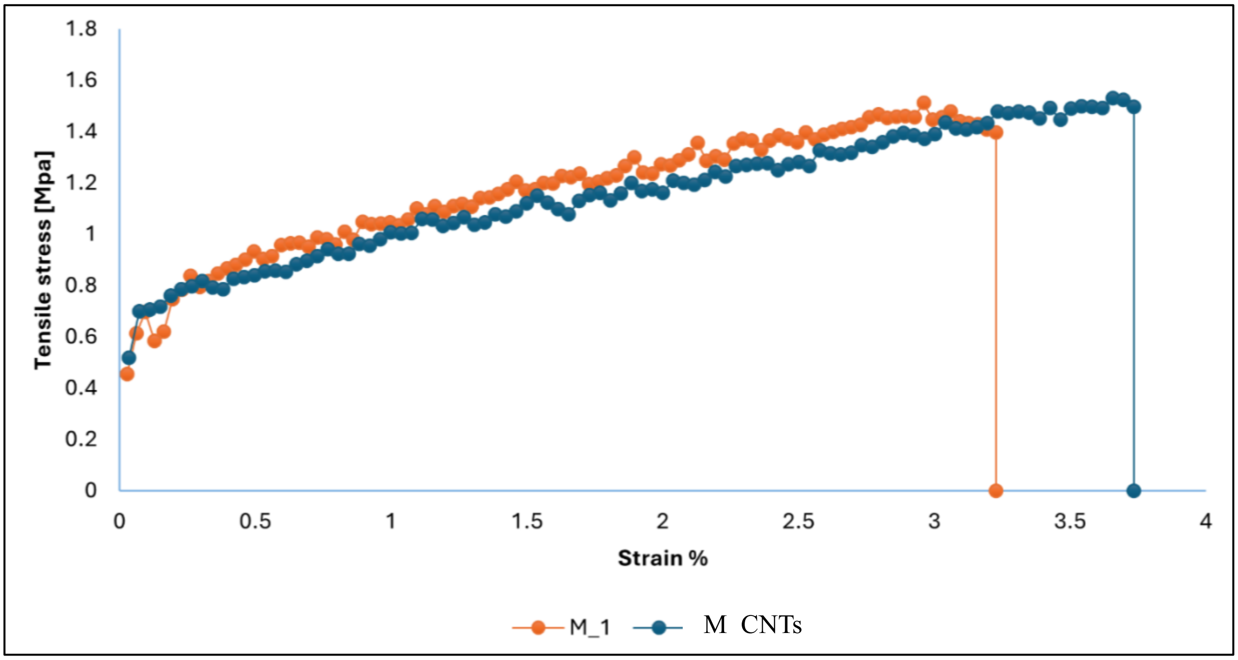

Fig 3.7: The mechanical properties of membranes

3.6 Performance of Membranes Test

To evaluate the efficient performance of the membranes, a simulation of the real operating system was performed as shown in Fig 3.8. using distilled water on an area of 0.00146 m^2 for both membranes, and the test was performed by fixing the pressure and time at different intervals to calculate the amount of flow, and the flow was measured several times to obtain better results as shown in Table 3.3 and Table 3.4. Fig 3.9 and Fig 3.10, which graphically represent the flow rate.

The performance of membrane in terms of mass flow and permeability was revealed by the Equations:

$$J_{W} = \frac{\Delta v}{A \, \Delta t}$$

$$Permeability = \frac{Jw}{P}$$ [Flux per pressure unit (bar)]

where, J_W is J is water flux, LMH , Δv is the of net water volume in Liter , A is an active area of membrane in m^2 , Δt is the time in hours , and P is Pressure.

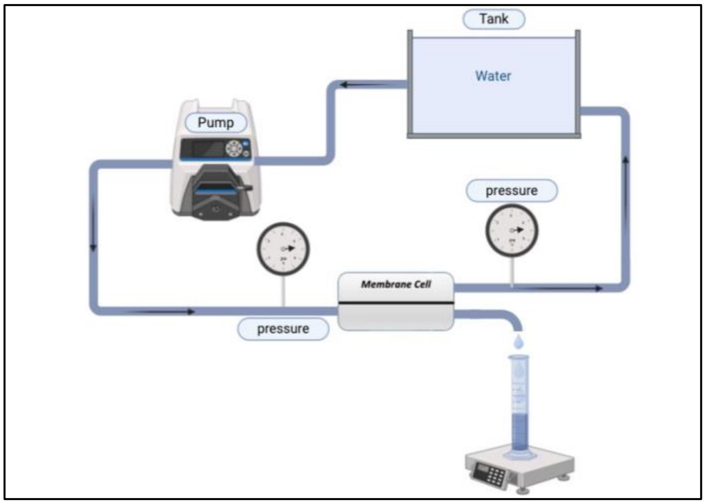

Fig 3.8: Membrane Efficiency Performance System

Table 3.3: performance membrane test for M_1

M_1 (Distilled water)							
The membrane is without MWCNTs							
Pressure (bar)	***Time (min)***	***Time (h)***	***Volume (g)***	***Volume (L)***	***Area(m^2)***	***J(L/h)***	***Permeability***
1	5	0.0833	3.4	0.0034	0.00146	0.19406	0.1941
	10	0.1667	6.56	0.00656		0.37443	0.3744
	15	0.25	9.46	0.00946		0.53995	0.5399
	20	0.3333	12.47	0.01247		0.71176	0.7118
	25	0.4166	13.5	0.0135		0.77055	0.7705
2	5	0.0833	4.2	0.0042		0.23973	0.1199
	10	0.1667	7.4	0.0074		0.42237	0.2112
	15	0.25	10.23	0.01023		0.58390	0.2919
	20	0.3333	13.5	0.0135		0.77055	0.3853
	25	0.4166	15.4	0.0154		0.87899	0.4395
3	5	0.0833	6.5	0.0065		0.37101	0.1855
	10	0.1667	8.3	0.0083		0.47374	0.2369
	15	0.25	11.2	0.0112		0.63927	0.3196
	20	0.3333	14.3	0.0143		0.81621	0.4081
	25	0.4166	16.5	0.0165		0.94178	0.4709

Table 3.4: performance membrane test for M_CNTs

M_2 (Distilled water)							
The membrane is with MWCNTs							
Pressure(bar)	***Time (min)***	***Time (h)***	***Volume(g)***	***Volume(L)***	***Area(m^2)***	***J(L/h)***	***Permeability***
1	5	0.0833	3.4	0.0034	0.00146	0.19406	0.19406
	10	0.1667	6.56	0.00656		0.37442	0.37443
	15	0.25	9.46	0.00946		0.53995	0.53995
	20	0.3333	12.47	0.01247		0.71175	0.71176
	25	0.4166	15.58	0.01558		0.88927	0.88927
2	5	0.0833	5.22	0.00522		0.29795	0.14897
	10	0.1667	9.83	0.00983		0.56107	0.28054
	15	0.25	13.84	0.01384		0.78995	0.39498
	20	0.3333	17.78	0.01778		1.01484	0.50742
	25	0.4166	21.61	0.02161		1.23344	0.61672
3	5	0.0833	5.52	0.00552		0.31506	0.15753
	10	0.1667	10.87	0.01087		0.62043	0.31022
	15	0.25	16.1	0.0161		0.91894	0.45948
	20	0.3333	21.1	0.0211		1.20434	0.60217
	25	0.4166	26.78	0.02678		1.52853	0.76427

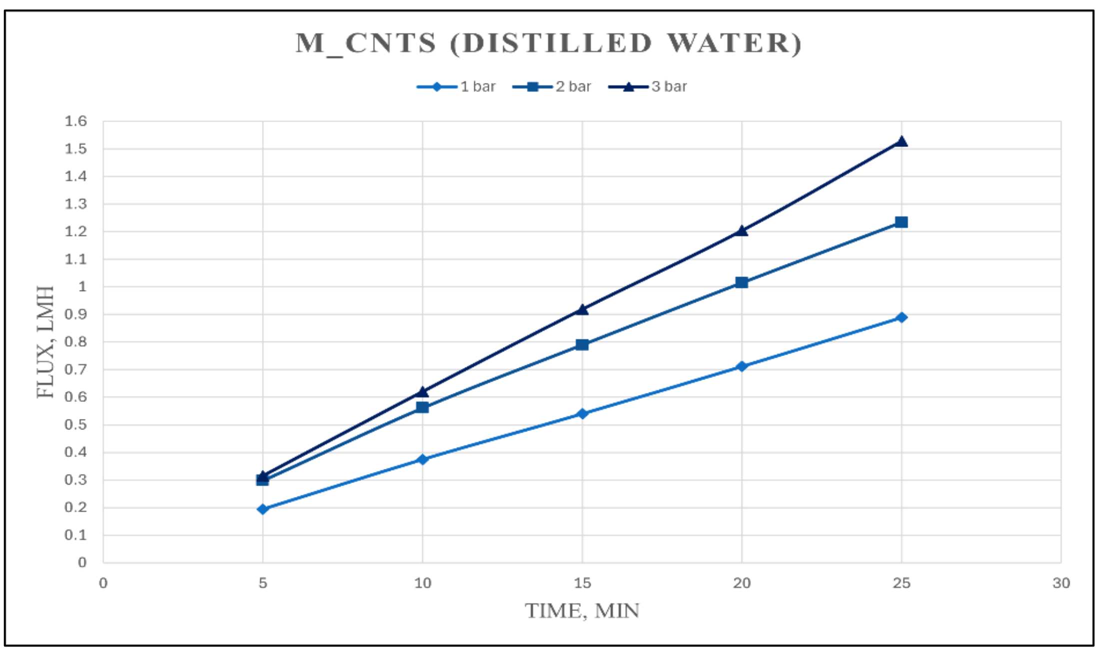

Fig 3.9: Mass flux per time for M_CNTs

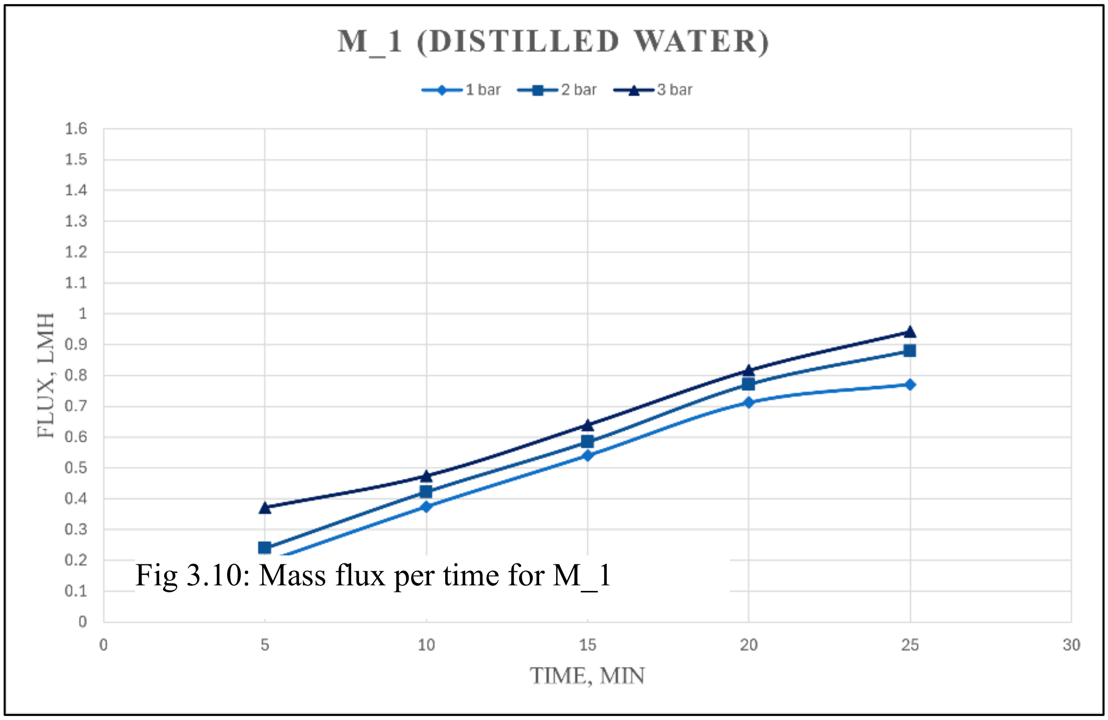

Fig 3.10: Mass flux per time for M_1

To find the total permeability of both membranes, the arithmetic mean was calculated for each of the three pressures, $Pe_{avg} = \frac{Pe1+Pe2+Pe3+Pe4+Pe5}{5}$.

The permeability enhancement of the MWCNTs membrane relative to the pure membrane is determined using, $P.E\ (\%) = \left|\frac{M\_1 - M\_CNTs}{M\_1}\right| \times 100$, where Pe is the permeability.

and the enhancement was 48.6% .The overall flow optimization percentage was calculated with the same steps as before using, $P.E_{avg}(\%) = \frac{P.E\ 1+P.E\ 2+P.E\ 3}{3}$.

and the overall flow optimization percentage was 49.82%.

CONCLUSION

Water scarcity is a global challenge exacerbated by population growth, climate change and industrial development. To overcome water scarcity, significant investments have been made in seawater desalination, water distribution, sanitation, and wastewater treatment. In this project modifying Polysulfone membrane (PSF) by using multi-walled carbon nanotubes (MWCNTs) and nanocomposites for water treatment applications. Two membranes were prepared, a pure Polysulfone membrane without any addition and a Polysulfone membrane with 1% MWCNTs. These two membranes were tested to determine the effects of different parameters on the membrane, as well as to evaluate the performance of the membrane efficiency in the desalination process using ultrafiltration, i.e. to obtain more flux and permeability.

Characterization processes were done using contact angle, SEM, TGA, and mechanical properties. TGA results showed that the MWCNT-added membrane has better thermal durability than the pure membrane. The mechanical properties results showed that the MWCNT-added membrane has better durability than the pure membrane.

Based on previous tests, the membranes were found to be suitable for conducting a membrane system efficiency test. The results of the system test showed that the new addition of MWCNT was effective in improving the membranes which led to an improvement in the flow rate and water permeability. The water flux of the MWCNT-optimized membrane is 49.82% relative to the pure membrane, while the water permeability improvement of the MWCNT-optimized membrane is greater than that of the pure membrane by 48.6%.

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