Polymer Composite CNT/Chitosan Nanofiltration Membranes for Water Desalination

By
Momina Batool

School of Chemical and Materials Engineering
National University of Sciences and Technology
2022

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Momina Batool
NUST-2018-274725MSE13SCME

This thesis is submitted as a partial fulfillment of the requirements

for the degree of

MS in Material Science and Surface Engineering
Supervisor Name: Dr. Nasir M. Ahmad

School of Chemical and Materials Engineering (SCME)
National University of Sciences and Technology (NUST)
H-12 Islamabad, Pakistan
August, 2022

Dedication

I dedicate this thesis and my entire education to my father Muhammad Ayub, my mother Saeeda Bano (late), my husband Muhammad Ali and my siblings Amna Ayub, Zain Ayub and Arfa Ayub for their support throughout my journey…

Acknowledgements

All the praises are attributed to the Almighty ALLAH the compassionate, the merciful. He is the source of all wisdom, who gave me strength to work for this thesis.

My major source of inspiration has been my supervisor **Dr. Nasir M. Ahmad** for his guidance and encouragement not only in preparation of this thesis but also in enhancing my learning in this area. His skills in reviewing my work and his suggestions contributed a lot in improving my work standards and for providing necessary laboratory facilities.

Support of RIC and HEC NRPU project No. 60202 is also acknowledged.

I want to thank my family for the patience and sincerity they showed. I thank my father, my mother (late) and my husband **M. Ali** who generated radiance to work hard and for their prayers towards my studies. I am also indebted to Dr. Aftab Akram and Dr. Mohsin Saleem. Their dedicated professionalism and enthusiasm for work has been a driving spirit behind the preparation of this thesis. I would like to extend my heartfelt thanks to Dr. Asim Laeeq Khan (Chemical Engineering Department, COMSATS, Lahore) for their cooperation and support. I express my deep gratitude to Ir. Dr Amir Azam Khan (Principal SCME) for their efforts to keep the environment congenial for research and study.

I want to thank my friends Amber Salim, Sana Shahid, Majida Chaudhary and Memonah Faryal for their emotional support throughout this work.

Mention needs to be made of all the seniors specially M. Asad Abbas, Dr. Mehwish Batool and Imran Ahmad Khan for valuable discussion and their time-to-time cooperation.

Regards,
Momina Batool

Abstract

Rapid population growth and industrialization throughout the world are creating a serious problem of access to clean drinking water. This makes the search for water treatment solutions effective and efficient. Removal of salt from contaminated water remains a significant challenge. In consideration of above, present study aims to develop nanofiltration polyethersulfone membranes using the phase inversion technique by incorporating novel combination of chitosan and carbon nanotubes to enhance salt rejection. The various membranes were fabricated including pristine PES membrane, PES membrane with chitosan (0.75 wt. %), with carbon nanotubes (0.1 wt. %) and CNT/chitosan composite in (0.1 wt. %) concentration composition. The membranes were further analyzed using attenuated total reflection–fourier-transform infrared spectroscopy (ATR-FTIR) for functional group analyses, scanning electron microscopy (SEM) for morphology and cross-sectional view of membranes, atomic-force microscopy (AFM) for surface roughness studies, water retention for water uptake analysis, mechanical testing for strength of membranes, and contact angle for water wettability. For membranes with the chitosan/carbon nanotubes composite, Scanning Electron Microscopy micrographs showed higher level of porous channels to render a better permeability potential, repeated by the flux rate results. Response surface methodology model was used to investigate the effect of feed temperature, feed concentration and feed pressure on flux and salt rejection. The salt rejection rates for the polymer CNT/chitosan were remarkable for Na_2SO_4 salt removal. The flux rate and salt rejection were also assessed for Na_2SO_4, and the results showed an increase from ±103 L/m^2h to ±436 L/m^2h for water flux and the salt rejection up to 95%. The increase in values of water uptake from ±54.39% to ±91.2% and decrease in contact angle from ±77.74° to ±61.26° showed an increase in the hydrophilic character of the membrane. The observed results are promising and provide an opportunity to treat water to remove contamination to improve the quality of drinking water.

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List of Abbreviation

CNT	Carbon nanotubes
NMP	N-methyl-2-pyrrolidone PES Polyethersulfone
WHO	World Health Organization
UN	United Nations
SEM	Scanning electron microscopy
ATR- FTIR	Attenuated Total Reflection–Fourier-Transform Infrared spectroscopy
Nm	Nanometer
C	Celsius
Ml	milliliter
µl	microliter
G	grams
Sec	Seconds
Rpm	Rotations per minute KBr
P	Membrane code for PES
PCH	Membrane code for PES incorporated with chitosan
PCN	Membrane code for PES incorporated with carbon nanotubes
P/CH/CN	Membrane code for PES incorporated with carbon nanotube/chitosan composite
AFM	Atomic Force Microscopy

Chapter No 1

Introduction

1.1 Background

Water is an important resource and the basis of all life present on this planet [1, 2]. It is a basic resource that should be available to all living things in the world. Although 72% of the Earth's surface is covered by water, only 0.5% is fresh water [3]. Figure 1.1 shows the distribution of water on Earth.

Oceans, as well as glaciers in the Antarctic and Arctic, and high mountain summits, are Earth's principal conventional water storage bodies. Groundwater accounts for 29.9% of all fresh water, while lakes, streams, and rivers store just 0.26 percent, and soil humidity accounts for 0.94 percent.

Water available for direct human use includes 96.80% groundwater, 0.02% runoff and 3.18% in lakes, while the main alternative sources of clean water are desalinated water and treated wastewater [3].

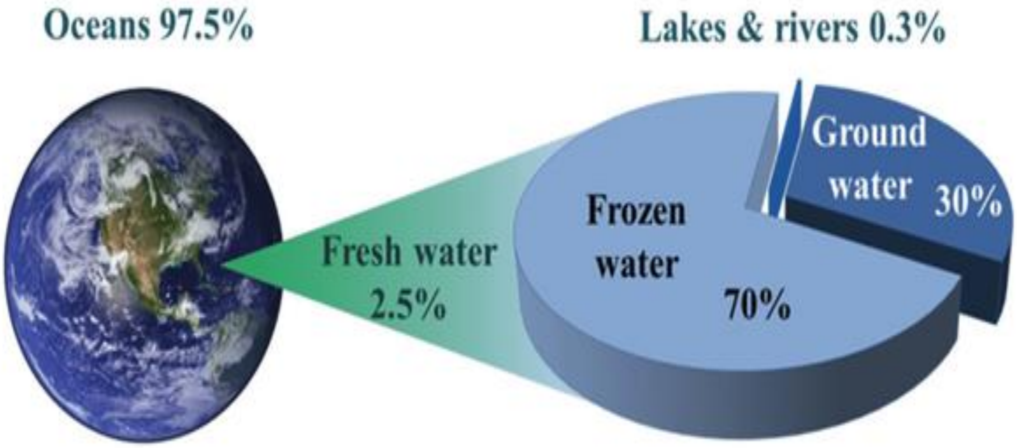


Figure 1.1 Earth’s Water Distribution

Water supplies are becoming inadequate due to high inhabitants' expansion rates, climate shift and water pollution. It is very crucial to solve the problem of water shortage. Combating water pollution is considered one of the main methods of solving the dilemma of water inadequacy [4, 5].

The most important international water disputes are the unequal division of water supplies, water-quality crises, worsening requirements and climate change over [6].Between 2015 and 2050, global population growth is expected to increase by 35%, leading to a rapid increase in water demand [7].

Although freshwater resources are abundant, the freshwater needed for human consumption only accounts for a small part of the earth's water supply [8]. In addition, when our water demand is increasing, the amount of fresh water remains constant [9].

Every year masses of people die due to lack of appropriate water supply and sanitation. Several resolutions have come about to aid among the ever-increasing water needs, including better management of water resources, reuse of water and desalination. Lots of the biosphere's rain-impacted conurbations have access to brine and groundwater, which can be converted to clean water by desalination to improve water needs[10].As a result, the world-wide need for salt removal is expanding, as evidenced with the steep growth of commercial water supply in recent years [11].

1.2 Pakistan's Water Crisis

Among many challenges associated to water contamination, public health is currently one of the most important problems. Pakistan's diverse water quality is far below the acceptable standard. Pakistan's drinking water quality is ranked number 80 out of 122 countries [12, 13].

Both ground and surface water are contaminated with dangerous metals, natural materials, pesticides, and other contaminants. Organizations in Pakistan do not follow the WHO's guidelines for safe drinking water. The trend of urbanization and industrialization has a significant impact on quality of life; as living standards have increased, it has a negative impact on the common environment, notably water resources. In Pakistan, significant water-borne illnesses such as cholera, hepatitis, diarrhea, and others are a big problem due to the use of contaminated water [14].

1.3 Polymeric Membranes: A Key to Water Security

Membrane technology is used to perform various activities and collection process challenges, and it is based on the same basic principle: membranes are utilized as a physical barrier between binary phases to filter particles, allowing fewer particles to flow through while retaining the residue[15].

Chlorination, membrane filtration, and chemical treatment are certain of the modern approaches for water disinfection/treatment. Boiling besides chlorination, on the other hand, entail a slew of other drawbacks. Chlorination produces harmful byproducts in the water, and the amount of chlorine left in the water must be managed before it can be used. Boiling necessitates a high temperature, and as water evaporates, the amount of water required decreases. Membrane technology has become a widely used approach for water therapy over time. To rally the assets of existing membranes, advancements appearing in membrane materials are necessary [16].

Membrane technology has fully-fledged greatly in popularity in modern years because of its modular design, low energy requirements, efficiency, and environmentally benign environment with minimal chemical use[17]. Membranes made of inorganic or natural materials have become more vital in water managing and water-related design. Membranes with various layouts are employed for specific applications all over the world, depending on their qualities and the material used [18].

Despite having several advantages over traditional water processes in terms of capacity and efficiency to perform various jobs, membrane technology has certain drawbacks in terms of water therapy, including flux slackening with time, fouling, hydrophobicity of polymers used, and so on. Particle microscopic filtration (e.g., viruses and bacteria) may not be efficient or complete due to changes in processing circumstances and arrangement composition [19]. Nanotechnology has played a critical role in overcoming these setbacks and improving performance by producing efficient membranes in recent years [18, 20–22].

Polymeric membranes have been successfully used for H_2O therapy in a variety of applications, including waste tributary against agro-food industries [23], petroleum industries [24], and materials or toxins deletion from drinkable water [25], allowing the distillate to be free or treated, thereby lessening the contaminations exclusively or tacitly discharged into effluent [26–28]. Reverse Osmosis (RO), Ultrafiltration (UF), Microfiltration (MF), and Nano-filtration (NF) exist as pressure-derived membranes that are considered promising opportunities for the expulsion of huge amounts of natural micro pollutants; however, RO and NF have been shown to be remarkably feasible filtration techniques in withdrawing micro pollutants [29, 30].

A membrane stands like a hurdle that permits certain particulars to pass through while obstructing others. Molecules, atoms, ions, and tiny particles are all possible candidates. The atomic membrane, natural membranes, and manufactured membranes are all different types of membranes. The concept of membrane creation has been used since the eighteenth century. Membrane technology is employed in a selection of life-sustaining processes. Polymeric membranes remain generally treated in water therapy because of their modest cost and ease of manufacture[31].

1.4 Problem Statement

Due to poor water supply and water-borne diseases claims millions of people's lives all around the world. In Pakistan, 40% of people die due to unsafe, unhealthy, and contaminated water. In developing countries frequently incidences occurring due to typhoid in middle and especially lower-class communities, the main leading cause of mortality and morbidity is due to water-borne diseases in Pakistan.

Water desalination is a major need nowadays. Efficient salt rejection membrane should be fabricated with enhanced flux and hydrophilicity to ensure the safe water contingency. The availability of high rejecting membranes to get safe pure water is the major issue of present times.

1.5 Research Framework

The research has been expanded on two major stages.

1.5.1 Stage I

Phase I includes successful synthesis of the membranes having different Additives. The polymer used is Polyether sulfone (PES) and Multiwalled Carbon Nanotubes, PVP and

Chitosan have been added as enhancers. The membranes were synthesized using these additives both separately and combined so that the effect of each additive can be understood better.

1.5.2 Stage II

In the second phase, different characterization techniques have been performed to investigate the outcome of additives on various properties. The synthesized membranes were characterized by SEM, gravimetric analysis, ATR-FTIR, mechanical testing, contact angle measurements, permeability flux and salt rejection, Water retention and Mechanical Testing.

1.6 Aims and Objectives

Following are main aims and objectives of research work

- Fabrication of polyether sulphone membrane
- Fabrication of polymeric CNT/Chitosan composite membrane
- Characterization of Fabricated membrane
- Aim of fabrication of composite membrane is to increase

1. Permeability flux
2. Hydrophilicity/ Wettability
3. Salt Rejection

Chapter No 2

Literature Review

2.1 Membrane Technology

Membrane is essentially a fine layer of partially permeable material that separates different substances when moving pressure is applied to it. Membrane procedures are widely used to remove particle matter, bacteria, microorganisms, and intrinsic organic material, and they can potentially give H_2O tastes, colors, and scents, as well as retaliate with germicides and bactericides, resulting in disinfection byproducts. It's a barrier that combines diffusion and sieving mechanisms to allow us to separate different species in a fluid. The aptitude of membranes to splitting particles and particles over a wide variety of molecular loads and particle sizes [32].

2.2 Strengths of Using Films in Water Treatment

Membrane techniques are used to provide the maximum quality water, regardless of the water source [33]. Membrane filtration, in contrast to other extra methods, removes various pollutants from the feed water, uses less chemicals, has a smaller environmental imprint, produces less precipitate, and is comparatively easier to maintain and run [34]. Membrane fouling, on the other hand, is a major flaw in membranes that restricts the improvement process. The sections below provide some data on membrane categorization and the different considerations that impact fouling.

2.3 Classification of Membranes

Pressure-directed membranes are classed into two groups [33].

- Low pressure Membranes
- High pressure Membranes

The optimal materials are thought to produce minimal fouling and are stable. Low pressure membranes and architecturally permeable membranes in which the pore size determines the separation process. Ultra-filtration and microfiltration are two different types of membrane filtration.

Microfiltration (MF) membranes bring out pore sizes stretching from 0.1 to 10 m, with the potential to turn down particles in the micron range. Ultrafiltration (UF) has a aperture size of around 0.01 m, which rejects tiny collides and viruses. Reverse osmosis and Nano-filtration belong to the second group of high-pressure membranes, in which the separation procedure is purely based on the solubility disparity between the solute and the solvent contained in the membranes [33].

Polymeric membranes and composites are fabricated compounds with a thickness of less than 1.0 mm that may be created from a variety of divisions and are affordable to produce under a variety of physical, chemical, and biological conditions. cellulose acetate (CA), polyvinyl fluoride (PVDF), and polyether sulfone (PES) are only a few examples of polymeric membranes in which the production material is either a synthetic or natural polymer. Water retention rate, surface charge, hydrophobicity, porosity, and roughness of the membrane material can all have an impact on membrane performance and fouling in different ways [35].

The contact angle reflects the link in the middle of the water and the membrane substance and is a gauge of membrane hydrophilicity. It has been determined that polymeric membranes are hydrophilic in nature, and that ceramic membranes are also hydrophilic [35]. While equated to ceramic membranes, polymeric membranes come up with a very strong negative charge and a neutral pH. Negative charge exists due to electrostatic repulsion when negatively charged compounds, such as NOM, are processed, which helps to decrease membrane fouling. Furthermore, smooth-surfaced membranes are extra vulnerable to fouling than rough-surfaced membranes. Pore size is important in low-pressure driven membranes because it determines whether particles will recruit through the membrane or not [36].

2.3.1 Membrane Morphology, Working Mechanism and Configuration

Membranes are classified as symmetrical or asymmetrical based on their structure. Membranes have symmetrical architecture, indicating that the penetrability of the membrane does not change as it passes out of the depth of the membrane. Asymmetric membranes, conversely, are made up of two layers: a fine functioning layer with low porosity and a very thin supporting layer (being-void space) that is extremely porous and aids in the production of hydraulic resistance. These structures can be used for high or low-pressure membranes. However, the configurations are different [37].

Flat sheet, hollow fiber, and tubular membranes are the most common designs in low-pressure membranes. Variable module parameters and manufacture have the greatest impact on fouling propensity [38]. High-pressure membranes are utilized for the spiral structure of the wound in widespread use, with a high packing density for the creation of bulk volume of water [39].

Membrane modules are reliant on membrane running processes. There exist two flow configurations in their operational processes:

- Drift in a dead end
- Purification by crossflow

Membrane flow is usually determined by fouling and the flux of permeate across the membrane. Membrane flow perpendicularly streams through a layer in dead end configuration, leaving several pollutant chemicals on the membrane surface [40]. The issue with this flow regime is that with reduced water output, the rate of fouling might increase [41].

2.4 Membranes

Membrane separation devices are extremely important in the separation market. Pressure-based membrane techniques were first employed in the water sector for reverse osmosis desalination in the early 1960s [42]. Separation in the liquid phase is based on four pressure-derived membrane systems: Ultrafiltration (UF), Microfiltration (MF), Nanofiltration (NF), and Reverse Osmosis (RO). These methods distinguish based on hydraulic pressure, which acts as a dragging force for flow transfer.

Furthermore, depending on chemical affinity, molar masses, particle size, and contact with membrane, membrane properties influence the flow of components selectively [41]. Figure 2.1 show the different kinds of membranes used on behalf of water treatment and their pore size range. Table 2.1 shows the symbols, pore size, operation pressure and materials removed by four filters.

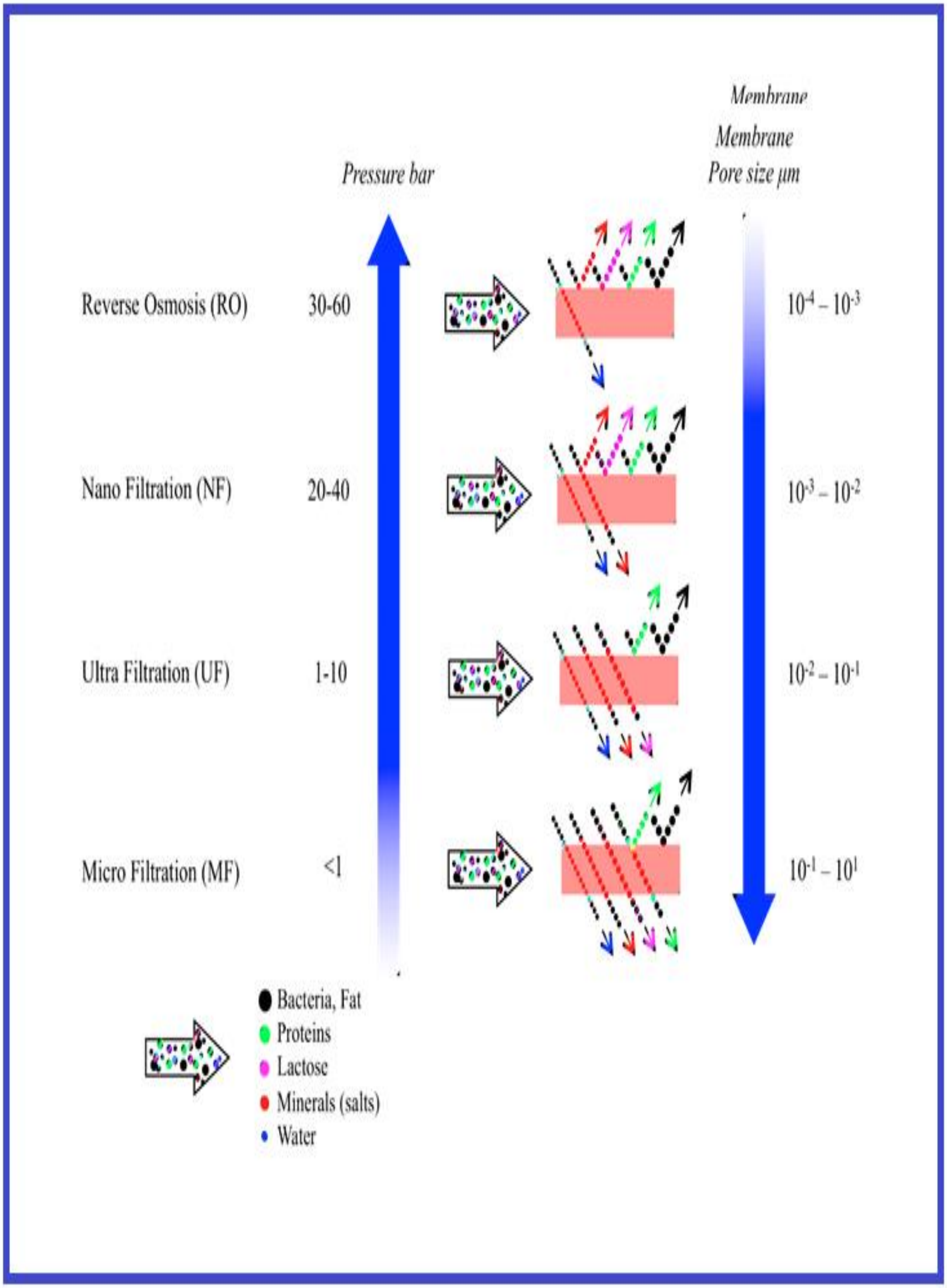
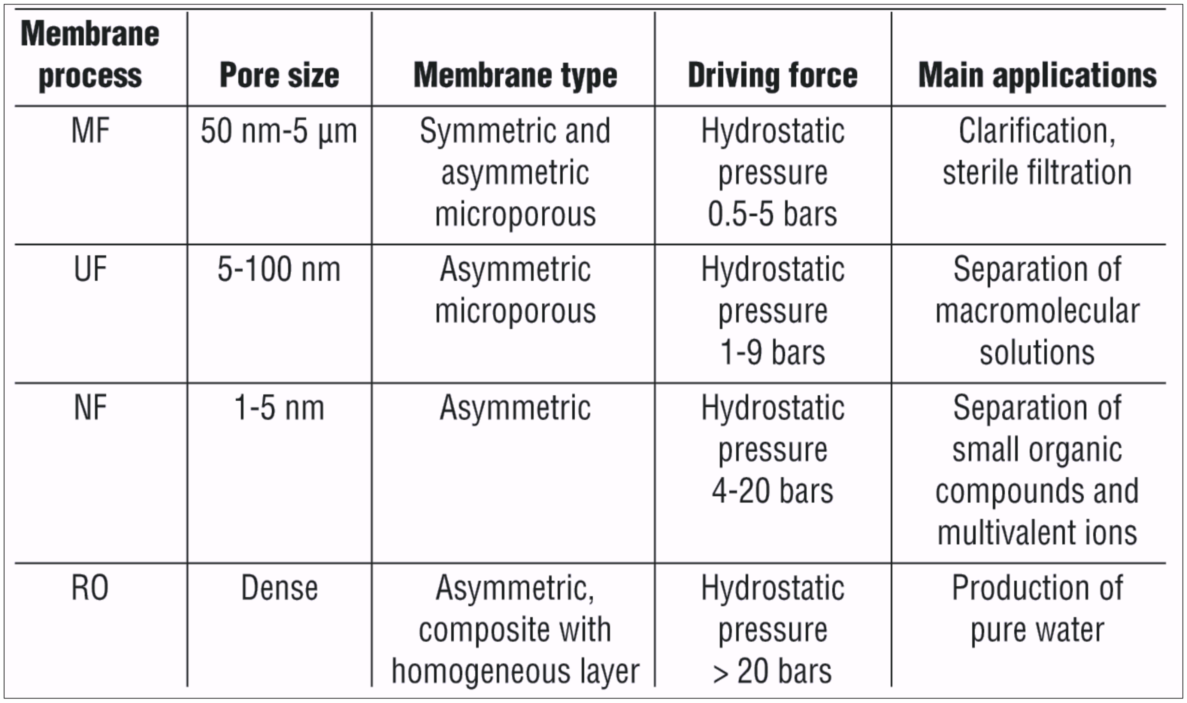


Figure 2.1 Schematic showing different categories of films used for water treatment and their pore size variety

Table 2.1 Illustrates the symbols, pore size, operation pressure and materials removed by four filters



Membrane process	Pore size	Membrane type	Driving force	Main applications
MF	50 nm-5 µm	Symmetric and asymmetric microporous	Hydrostatic pressure 0.5-5 bars	Clarification, sterile filtration
UF	5-100 nm	Asymmetric microporous	Hydrostatic pressure 1-9 bars	Separation of macromolecular solutions
NF	1-5 nm	Asymmetric	Hydrostatic pressure 4-20 bars	Separation of small organic compounds and multivalent ions
RO	Dense	Asymmetric, composite with homogeneous layer	Hydrostatic pressure > 20 bars	Production of pure water

2.4.1 Microfiltration (MF)

Microfiltration is a type of filtration that removes molecules and transforms them into a microporous membrane (with pores ranging from 0.05m to 10m). Furthermore, the pressures used in this procedure are less than 0.2 MPa. The pore size of microfiltration membranes limits from 0.1 to 10µm. Biotechnology (cell isolation using fermentation broth), the food/dairy sector, and the treatment of latex and oil emulsions are only a few of the fields where MF is used [43–49] . It's also utilized in the pharmaceutical business for protein filtration (to remove DNA or viruses) and plasma separation in blood therapy [50–52]. Anoxic pond effluent treatment, municipal wastewater reclamation, and toxic chemical removal from drinking water are some more instances of its utilization[53].

2.4.2 Ultrafiltration (UF)

Microfiltration membranes have traditionally been described in terms of pore size (in µm), whereas Ultrafiltration membranes are described in terms of the molecular mass of the elements that the membrane aperture can inhibit. Isolating particles with molar masses ranging from 1 to 300kDa requires a pressure greater than 1MPa[50]. Suspended particles (solids and solutes) having a molecular weight larger than 300 kDa are rejected, whereas molecules with a lower molecular mass and water pass through the membrane. UF is used in the production of drinking water, food refinement[54] and the separation of undesirable proteins in food and dairy products[55–57]. In addition, it has uses in biotechnology, such as harvesting cells, liposomes, and lysozyme [58]. Furthermore, important pollutants can be recovered from waste streams employing this method[59].

2.4.3 Nanofiltration (NF)

Nano filtration is a membrane-based separation method that separates wastes and dissolved compounds based on their molecular mass (between 350 to 1000 Da). This procedure requires pressures ranging from 4 MPa to 20 MPa [52, 60]. NF is a relatively new membrane filtering tool, having been created in the mid-1980s [59]. The softening of hard water by eliminating polyvalent cations from groundwater aquifers and surface water is some of the extremely critical functions of NF. This technique has recently been claimed to have been employed to eliminate byproducts, such as natural and synthetic materials [61, 62]. It's also approved for food processing applications like coffee separation, oligosaccharide extraction, and green bio refineries[63].

2.4.4 Reverse osmosis (RO)

In contrast to microfiltration and ultrafiltration membranes, the reverse osmosis membranes are dense and lack identifiable holes. This technique is similarly pressure-dependent (varying from 20 to 80 MPa) and can remove monovalent ions (350Da) and small pollutants from solutions [41]. Because of size inclusion, the solution's diffusion process [64], and charge limitation, physical or chemical interaction within the solutes, solvent, and membrane, mass transfer is feasible in RO. Reverse osmosis has the potential to purify seawater into drinking water by removing salts and other contaminants. In the elimination of industrial effluent from diverse chemicals, pulp & paper, food items, and metal finishing industries, the efficiency of this approach has remained high. Nonetheless, this system's input water requirements are very stringent in terms of suspended elements such as particles, fibers, and oily substances[63][42]. When RO and UF are combined with distillation, per evaporation, and other separation processes, a hybrid system with selective operations and high efficiency is created. The technique's broad variety of applications assisted in the choice of an acceptable membrane design and material, allowing numerous criteria such as membrane configuration, chemical structure and stability, and ease of manufacture to be considered [42]. Figure 2.2 show a comparison between different types of membranes against 2D material NF membrane.

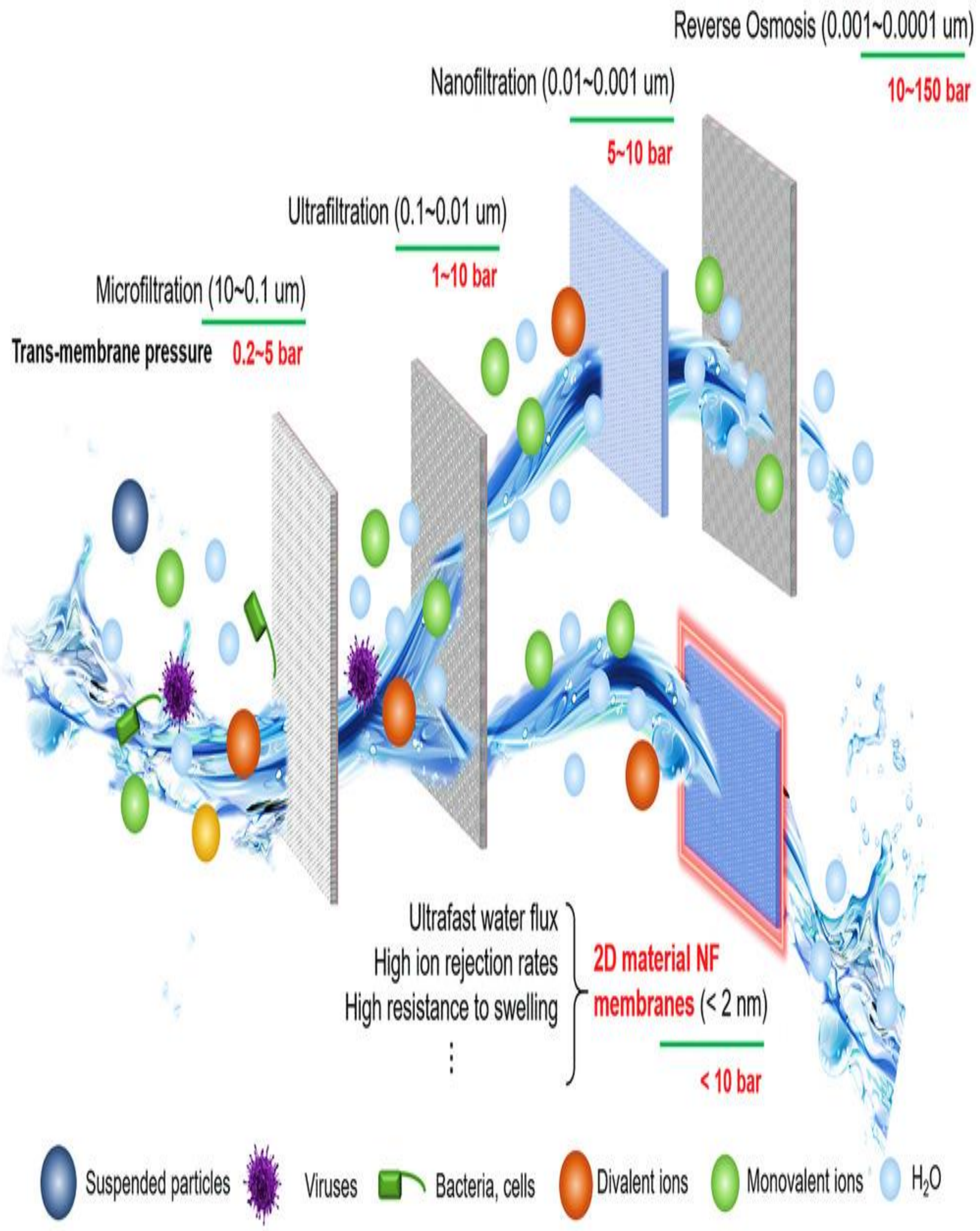


Figure 2.2 Different types of membranes against 2D material NF membrane

2.5 Polyether Sulfone (PES) Membranes

A wide range of nanomaterials are used in the membrane fabrication process. While synthetic organic polymers such as cellulose acetate, nylons, polyether sulfone (PES), polyvinylidene fluoride (PVF), polysulfone, and others are used in the production of ultrafiltration and microfiltration membranes, polyetherfulsone is well-recognized for its ultrafiltration membrane production [65]. In the production of polymeric membranes for H_2O treatment PES is acknowledged as a key polymer. It's commonly available on the commercial market, and it's typically manufactured by connecting two functional groups (the ether and sulfone groups) to the backbone. Figure 2.3 depicts the PES structural formula. These polymers have numerous desirable qualities when employed in membrane fabrication, including hydrolytic stability, high dimensional stability, high thermal stability, and outstanding oxidative properties, among others [66]. Figure 2.4 depicts an image of PES powder.

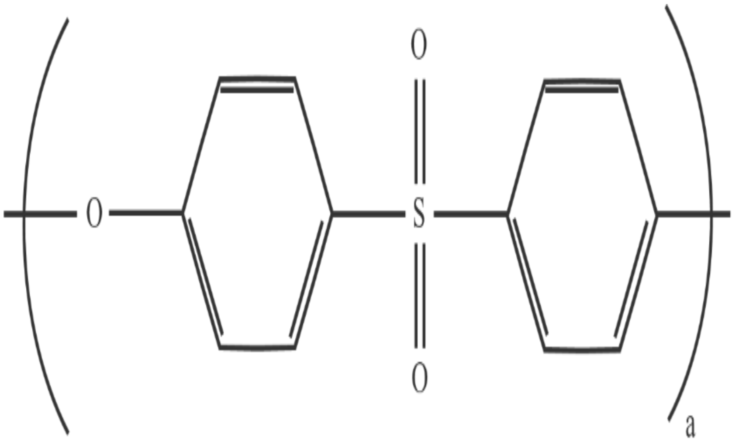


Figure 2.3 Structural Formula of Polyethersulfone

One of the noteworthy limitations of this extraordinary polymer [107] is its aquaphobic nature. The hydrophobicity of PES membranes reduces the membrane diversity and makes them more prone to membrane fouling [67]. The phase inversion approach of producing polyether sulfone membranes results in an asymmetrically formed membrane. The features and appearance of a fabricated membrane are influenced by a variety of elements such as additives, polymer strength, solvent, casting temperature, and so on [66]. PES membranes are now subjected to various alterations to increase performance[66–68].

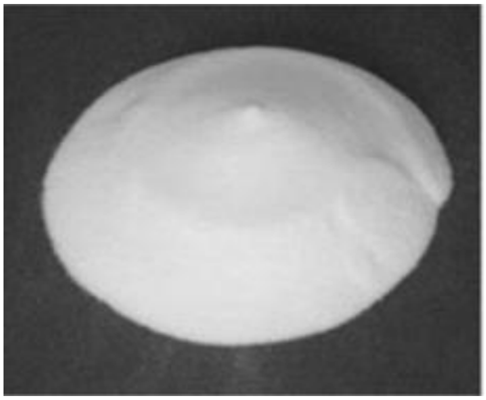


Figure 2.4 Polyethersulfone powder form

2.6 Chitosan

Chitosan is made up of a linear polysaccharide chain with a 1, 4-glycosidic bond that is produced by incomplete deacetylation of chitin, a crucial constituent of crustacean shells (e.g., crab, shrimp, and crawfish). Repetitive units of chitosan include glucosamine and N-acetylglucosamine due to incomplete deacetylation. Because of its unique chemical and biological features, this molecule is extremely important [69]. Because chitosan includes a lot of amino (–NH2) and –OH functional groups in its composition, it has a lot of reactivity as shown in Figure 2.5.

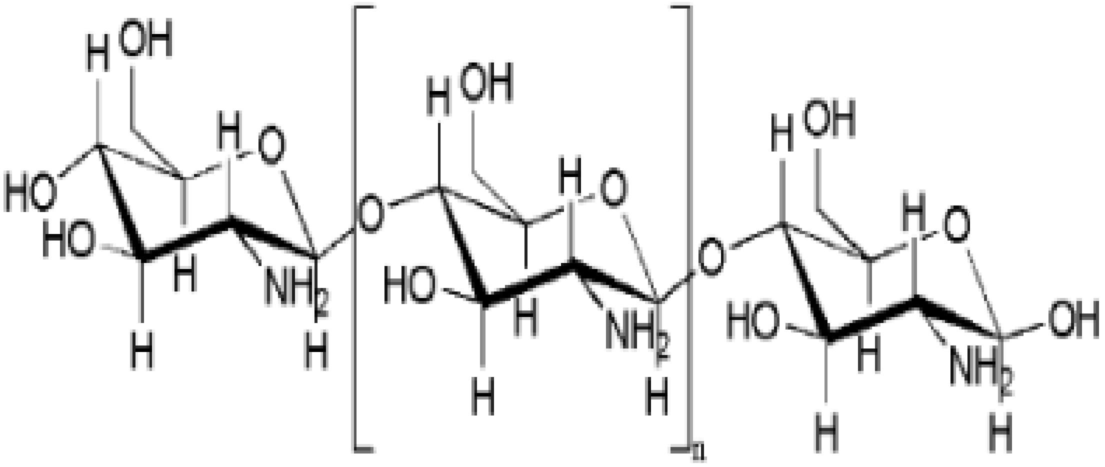


Figure 2.5. Structure of completely deacetylated chitosan (Reproduced from reference [[73]).

Chitosan is soluble in aqueous acids, but inorganic solvents are insoluble [70, 71]. When the pH is below 7, the amino groups can be protonated, resulting in the formation of chitosan, a water-soluble cationic polyelectrolyte. When the pH of chitosan exceeds 7, the amino groups become deprotonated and insoluble. The adding of several functional groups that can form covalent bonds with chitosan can alter the characteristics of chitosan (i.e., the degree of hydrophobicity can be altered). Chitosan, on the other hand, can be made into gels, films, beads, and fibers [72].

2.7 CNT materials

Carbon nanotubes (CNTs) are one-dimensional similarities of zero-dimensional fullerene molecules that have a cylindrical carbon network. A nanotube is a micrometer-scale graphene sheet that has been turned into a nanometer-scale cylinder and overlaid with a globular fullerene (Figure 2.6). In the x-y plane, graphene layers are made up of a monolayer of sp^2 - bound carbon atoms. CNTs exhibit unique electrical characteristics owing to the presence of a delocalized - π- electron in the z-axis [74].

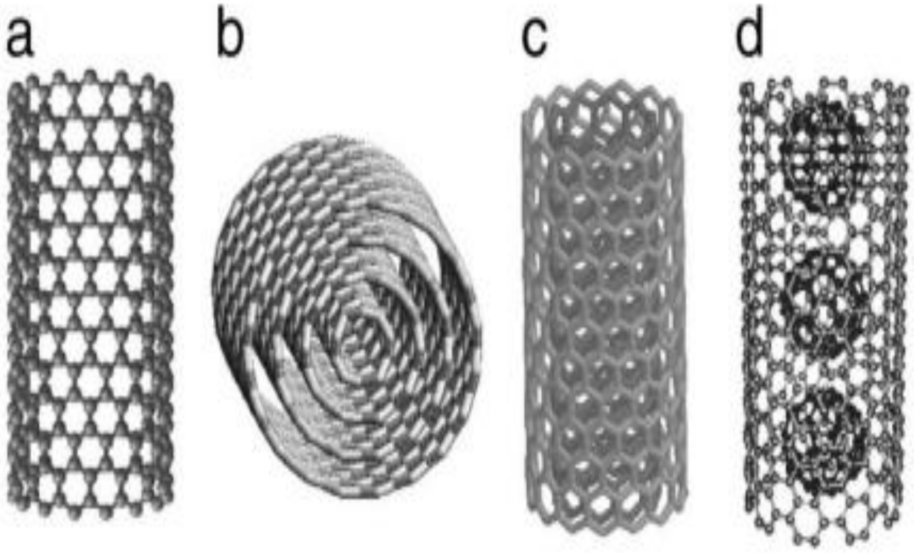
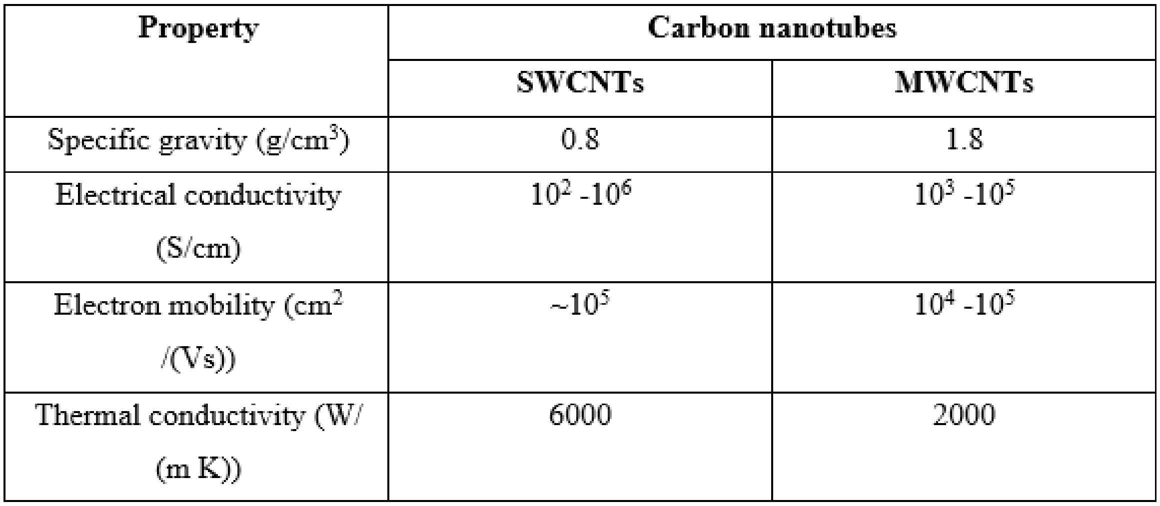


Figure 2.6 Graphic representations of (a) single-wall carbon nanotubes (SWCNTs), (b) multi-wall carbon nanotubes (MWCNTs), (c) double-wall carbon nanotubes, and (d) peapod nanotubes made of SWCNTs packed with fullerenes [77].

The two major types of carbon nanotubes are single-wall carbon nanotubes (SWCNT) and multi-wall carbon nanotubes (MWCNT) (Figure 2.6). In the same way that a cylinder is built up of benzene units with hexagonal rings and double and single carbon-carbon linking, SWCNTs are composed of a two-dimensional array of benzene molecules. Many layers of coiled graphene sheets make up MWCNT. Depending on their chirality and diameter, SWCNT are either metallic or semiconducting nanowires [75, 76].

The properties of carbon nanotubes are concise in Table 2.2 [78]. The electrical conductivity of SWCNTs is higher than that of MWCNTs (102-106 S/cm). Thermal conductivity of SWCNT is three times that of MWCNT.

Table 2.2 Physical properties of CNTs [78].



Property	**Carbon nanotubes**	
	SWCNTs	**MWCNTs**
Specific gravity (g/cm^3)	0.8	1.8
Electrical conductivity (S/cm)	10^2 -10^6	10^3 -10^5
Electron mobility (cm^2 /(Vs))	~10^5	10^4 -10^5
Thermal conductivity (W/ (m K))	6000	2000

The strong attractive forces flanked by CNT and the weak inter-planar contacts of the graphene sheet remain are caused by the high polarized -electron clouds in CNT. Individual nanoparticles' physical properties, for instance size of it, form, and superficial area of carbonaceous nanomaterials, are heavily influenced by accumulation state along with solvent chemistry. Secondary structure physicochemical assets of nanomaterial masses are highly varied and poorly defined [79]. For carbonaceous nanoparticles to be used in water purification-membrane filtration, these features must be resolved [80].

CNT is hydrophobic by nature, and this feature helps in water purification. For starters, hydrophobicity and capillarity have a role in sorbate adsorption and orientation in microporous carbons. For unfunctionalized nanomaterials, physio-sorption is the most common sorption mechanism. Surface-assimilative ability has significant consequences for pollutant removal and hydrogen storage in environmental applications. For the amputation of an organic in addition to inorganic contamination, such as NOM and heavy metals, CNT for traditional drinking water treatment relies heavily on physicochemical sorption processes [81–83].

Carbon nanotubes (CNTs) have piqued the interest of scientists for a variety of uses throughout the last few decades [84–87]. Because of its features, such as high transport rates and narrow pore apertures, CNTs are mostly used to make membranes [88]. However, since carbon nanotube production is relatively overpriced and restricted to minor-scale fabrication, there are trials that restrict the usage of carbon nanotubes on a broader scale. Another issue beside carbon nanotubes is their hydrophobicity, which can border their use in membranes [87, 89].

CNTs can correspondingly exist when spread in a polymer solution, which could be intriguing way to make MMMs with a high mass transport rate. Furthermore, dispersion in a polymer can aid to minimize CNTs' hydrophobicity, creating them suitable for usage as membranes. Due to the ease of filtering the resulting dispersions to generate the membrane, which is also known as "buck papers," the sonication process is regarded one of the most effective means of scattering CNTs across a polymer [89]. While ultrasonic momentum is employed to disperse large amounts of CNTs, the resultant products are stabilized by non-covalent interactions with the polymer molecule (surfactant), according to several studies [90, 91].

Chapter No 3

Experimental work and Characterization Techniques

3.1 Materials

Throughout the experiment, analytical grade substances were utilized. The membrane casting procedure was carried out with distilled water, whereas the chitosan/CNTs solutions were made with deionized water. Polyethersulfone (58000 M_w) was acquired from Ultrasone, Germany. N-Methyl-2-Pyrrolidone was bought from Sigma-Aldrich, Germany. Polyvinylpyrrolidone (PVP, Mw 40,000 g/mol) was got from Merck, Germany. Multiwall CNTs (diameter 10-30 nm) were obtained from Merck, Germany.

3.2 PES with CNTs, chitosan and CNTS/chitosan Mixed Matrix Membrane

The experimental procedure involves formation of membranes with 17.5% wt. incorporated with chitosan, CNTs and chitosan/CNTs. A polyester fabric sheet was used as support for membranes casting.

3.2.1 Membrane Fabrication

Membranes were produced employing the phase inversion approach. Four diverse membranes were fabricated i.e., PES with PVP, PES with additives and PES with composites, were all fabricated by same procedure. To prepare a casting solution, PES was added to NMP solvent as repeatedly stirring in a media bottle. For 24 hours, the solution was heated to 60° C in addition stirred at 250 rpm. As stated in the table, the casting mixtures were made up of various blends. PES, PVP as a pore forming, chitosan, and carbon nanotubes were all used.

3.2.2 Composite chitosan/CNTs nanoparticles preparation:

20mL of a 1% (v/v) acetic acid solution were mixed for an hour to evenly distribute 0.1% of the necessary amount of CNTs. The nanoparticle aggregation was then dispersed by ultrasonic for 30 minutes at room temperature. The solution was then mixed while 0.75% chitosan powder was added, and it was sonicated for a further 30 minutes to completely dissolve the chitosan and combine with the CNTs. The reaction took place for two hours at 60 °C while being stirred. The finished product was then recovered via filtering, and the pH was adjusted to around 7 by washing with distilled water.

3.2.3 Nanocomposite Mixed Matrix Membrane Preparation:

17.5% w/w of total polymer solution was prepared. All the chemicals were properly weighed. 3.5g of PES was dissolved frequently in 12ml of NMP and stirred at 250rpm for 24 hours. 2.5 w/w% of PVP was added frequently stirring was continued for next 24 hrs. After the addition of additives, the solution was stirred for 120 hours.

Figure 3.1 Polymer membrane manufacture appliance (Automatic Film Coater)

The solution stood sonicated for 30 minutes and again heated and stirred for 30 minutes. The solution was kept for 2 hours for degassing of trapped air. It was casted on polymer membrane casting apparatus (Automatic Film Coater) at room temperature on a speed of 50–60 mm/s beside water as a non–solvent, and then added into the coagulation bath for 5 minutes and then dipped in water/isopropanol 70/30 solution for 19-20 hrs. The prepared membranes were further dipped in glycerol for 3-5 hrs. The synthesized membranes were the washed with distilled water then air dried and stored as shown in Figure 3.2. The composition of membranes is shown in Table 3.1

Table 3.1 Percentage Composition of membranes

Polymer Membrane	**PES %**	**PVP%**	**CNTs %**	**Chitosan %**
P	17.5	2.5	-	-
P/CH	17.5	2.5	-	0.75
P/CN	17.5	2.5	0.1	-
P/CH/CN	17.5	2.5	0.1	0.75

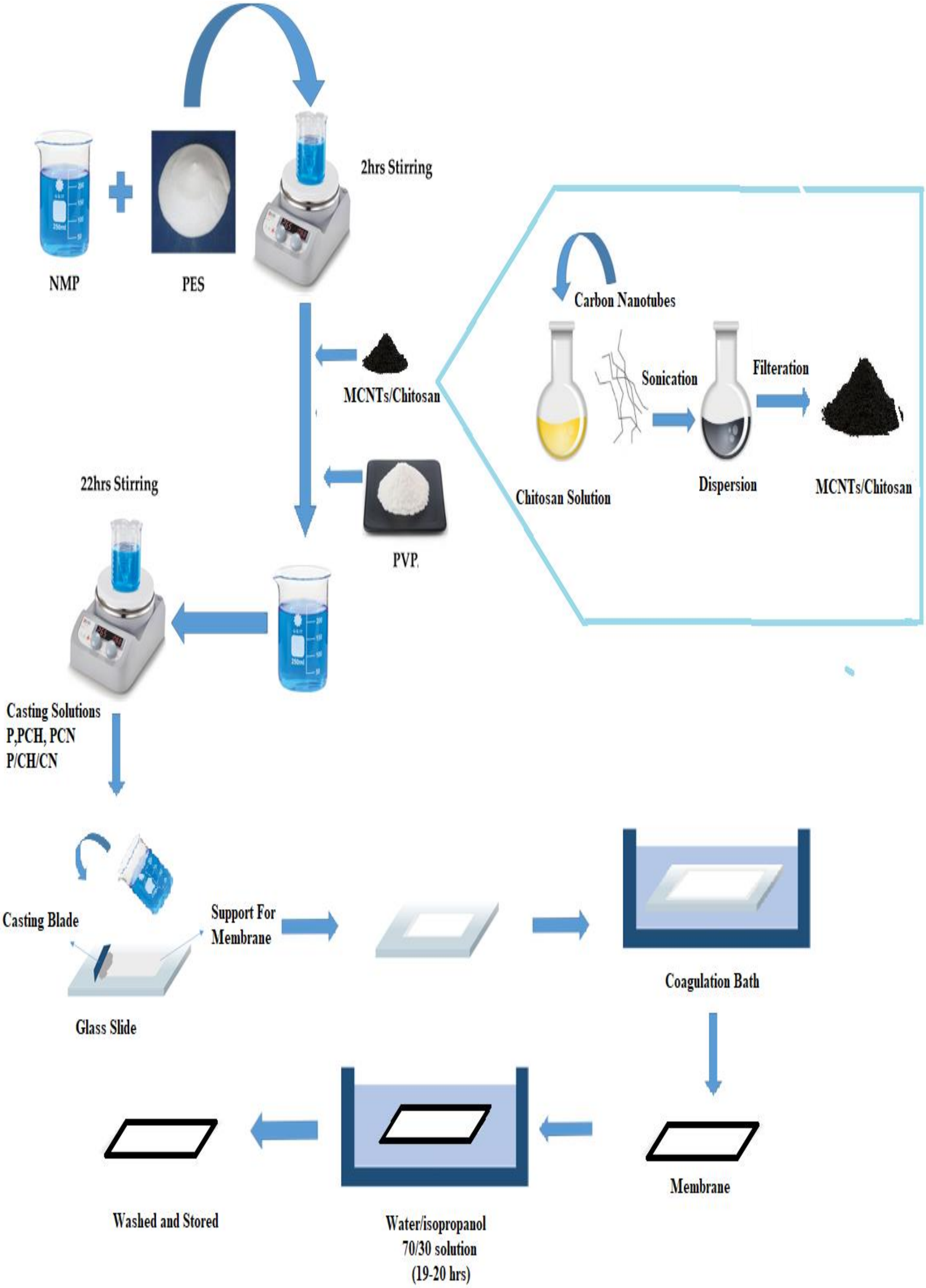


Figure 3.2 Schematics for membrane synthesis

3.3 Characterization Techniques:

3.3.1 Hydrophobicity and contact-angle measurement

The contact angle instrument was used to investigate the hydrophobicity attribute (sessile drop method). A deionized water droplet was injected into a dry membrane sample of around 1 cm^2 and a picture was obtained. For the lowest error, the angle amongst the membrane surface and the droplet was assessed three times arbitrarily. This was accomplished using the KRUSS DSA-25 drop shape analyzer.

3.3.2 Swelling measurements

Membrane samples were dried and sliced into thin strips (usually 4 mm x 10 mm). A digital balance was used to determine the weight of every single sample. The samples were submerged in distilled water for 24 hours at 21°C. At regular intervals, the strips were removed, weighed, and positioned between two filter sheets to remove extra liquid. The percentage extent of swelling (SW) was determined using the following formula:[106]

$$\mathbf{SW}= \frac{W_S - W_o}{W_o} \qquad \textbf{Equation (1)}$$

Where W_s is the weight of swollen membrane and W_o is the weight of dry membrane.

3.3.3 Porosity

The membranes' bulk porosity was calculated using gravimetric methods. Three membranes were sliced and submerged in distilled water at room temperature for 24 hours. The wet membranes were then weighed after being placed between two filter sheets to remove any leftover solvent on the membrane surface (W_w). The wet membranes were then dried in an oven at 50°C for 2 hours before being weighed to determine their dry weight (W_d). Membranes were assessed for thickness. Using the following formula, the porosities of the produced membranes were determined using the dry-wet method.[107]

$$\boldsymbol{\varepsilon}= \frac{W_w - W_d}{Vd} \qquad \textbf{Equation (2)}$$

Where W_w and W_d stand for the wet and dry weights of the membranes, d water is the density of pure water at 21°C, and V is the volume of the membrane in the presence of water.

3.3.4 Scanning Electron Microscopy

The membrane shape and surface structure were studied using SEM. SEM was performed with a JEOL-JSM-6490LA, which had a working distance of roughly 10 mm, an operating voltage of 10–20 kV, and a spot size of 35–60. The membranes used were sliced into 1 cm^2 pieces and frozen in liquid nitrogen meant for dehydration in addition to breaking into tiny pieces without troubling the cross section.

3.3.5 FTIR

The functional groups of the membrane were studied using FTIR. For ATR-FTIR, the dry membranes were sliced into 0.5 cm^2 diameters. The resolution was roughly 2 cm^{-1} and the spectral range was between 400 and 3500 cm^{-1}. The experiment occurred on a BRUKER ALPHA II FTIR spectrophotometer.

3.3.6 Mechanical testing

Membrane samples were sliced into dog-bone shapes according to ASTM D882. The mechanical properties of membranes were examined using a tensile tester, the Universal Testing Machine (UTS), at a regulated temperature of 21°C (Shamizdu AG-X Plus). Five sample strips from each membrane were checked prior to testing, each measuring 25 mm by 3 mm with a thickness of 0.3 assessed using a micrometer. As shown in the figure, the samples were tested at a strain rate of 0.5 mm/min with a gauge length of 12.5 mm. The maximum stress of each sample was measured, and the slope of the linear component of the stress-strain curve was used to compute Young's modulus. At the break, elongation and tension were determined [108].

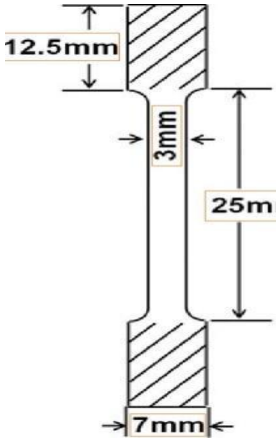


Figure 3.3 Dog-Bone style

3.3.7 AFM

A JEOL JSPM-5200 scanning probe microscope was used to display the results of atomic force microscopy (AFM) measurements on each of the produced membranes. A tapping mode was used to scan a 15m^2 area of the membrane surface to capture photographs of the membranes. The instrument software was used to acquire each average roughness value (Ra).

3.3.8 Flux and salt rejection

The amount of fluid moving through a membrane under influence of different parameters, also including time, area, volume, and so on, was studied to calculate the permeation flux of the membranes. The pressures of the filtration assembly in which the membrane specimen was held were varied. The schematic configuration for calculating the water flux is shown in Figure.3.4.

The time and volume of distilled water flowing through the membrane were recorded. The obtained data was then entered into Equation[109, 110]

$$\boldsymbol{J = \frac{V}{AT}} \qquad \textbf{Equation (3)}$$

Where V is the volume of water, A is the membrane's surface area, and T is the permeation flux time, J is the permeate flow computed in Lm^{-2}h^{-1}. Three values were computed for each membrane to ensure the least amount of inaccuracy.

Running of the made nanofiltration membranes was also examined by evaluating the rejection Na_2SO_4. Salt rejection efficiency (%SR) was computed using Equation (4) [111].

$$\%\mathbf{SR} = \mathbf{1} - \frac{\textbf{Conductivity of permeate } (\mathrm{C}_p)}{\textbf{Conductivity of feed } (\mathrm{C}_f)} \times \mathbf{100} \qquad \textbf{Equation (4)}$$

The conductivity of the feed and permeate solutions was determined employing a conductivity meter (Cyber scan PC 300 Series, Lahore, Pakistan). The feed solution was made with distilled water.

3.3.9 RSM

The performance of the membrane process was investigated using response surface methodology (RSM) established on the central composite design (CCD). To achieve accurate forecasts near the extremes of the factors, CCD was chosen.

Table 3.2 Factors and Range for design of Experiments

Code	**Factors**	**Units**	**Code Level**			
			Low	**High**	**-alpha**	**+ alpha**
A	Feed Concentration	(mg/L)	500	2500	181.793	3181.79
B	Feed Temperature	(°C)	25	40	19.8866	45.1134
C	Feed Pressure	(kg/cm^2)	2	6	0.636414	7.36359

The factors that were found as key parameters contributing to therapy were concentration, temperature, and pressure. For the experimental design, Design Expert software was utilized, and the levels of the factors are displayed in Table 3.2. Table 4.3 shows the design matrix generated following the application of CCD. Table 4.3 has a total of 20 sets of runs, including six level runs that are centrally coded. Design expert was used to create response surface plots to determine the effect of the input parameters on the responses.

Chapter No 4

Result And Discussion

4.1 FTIR

The ATR FTIR spectra in Figure 4.1 show most of the PES polymer's distinctive peaks. All membranes have peaks at 1580 cm1 (strong C=C bending intensity shows benzene ring) and 1485 cm^{-1} (C-C bond stretching), which are consistent with the Polyethersulfone structure [112].

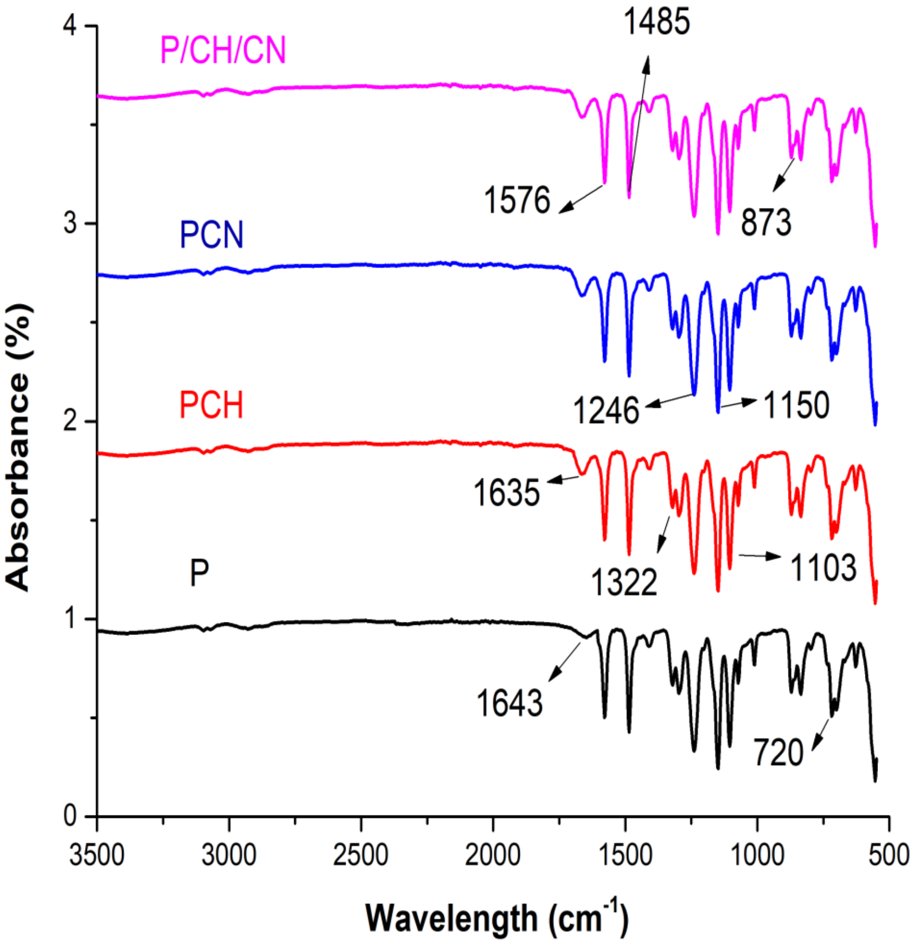


Figure 4.1 ATR-FTIR spectra for P, PCH, PCN, P/CH/CN membranes

For the composite membrane, these peaks may also be seen in P/CH/CN. C–O stretching of the ether and carboxylate structures is confirmed by bands visible at 1200 cm^{-1}. The existence of a sulfone group in the polyether sulfone structure, which is corroborated by literature, identifies the aromatic ether group as C–O–C stretching at 1246 cm^{-1} [113]. The sulfonyl (O=S=O) group is responsible for the peaks at 1150 cm^{-1}. C–O and C–H vibrations at 873 cm^{-1} suggest the presence of CNTs.

The –C–O–C– bonds match to the absorbance peak at 1103 cm^{-1}, showing the presence of chitosan. The methyl groups produce two weak bands at 1322 and 1395 cm^{-1} that are only found in the spectrum of polyether sulfone [114–119]. Since the polyether sulfone concentration is much higher than 0.75 and 0.1 wt.% of the additives, there is no noticeable change in the spectra after adding chitosan, CNTs, or a Chitosan/CNTs composite to any of the membranes in Figure 4.1 [120].

4.2 SEM

The top surface morphology as well as cross-section formation of the composite membrane were investigated employing SEM. As shown in Figure 4.2 the created membranes, the asymmetric morphological formation consists of a dense topcoat and macro-void forms at the lowermost, as depicted in Figure 4.2. The P and PCH show a dense top layer showing perfect mixing of polymer solution whereas PCN, P/CH/CN show the formation of agglomeration at the top [116].

The cross sections divide up of the membranes are depicted in the micrographs in Figure 4.2. These membranes devise an asymmetrical assembly with a gradient-like formation, as depicted. All the PES membranes have a dense top layer. A porous sublayer composed of finger-like structures known as channels is also visible. Permeability improves when channel connections and channel formation improve.

A squeegee-like mesoporous form can also be observed underneath this sub layer. The inclusion of chemicals like PVP along with chitosan causes a small alteration in the morphology. Because both PVP as well as chitosan have high molecular weights, they increase the viscosity of the solution, reducing the development of macro voids [121]. Not only does the addition of CNTs to the membranes enhance porosity, but it also increases surface roughness. The pores usually widen/expand when CNTs are added, resulting in greater membrane porosity[122]. PVP and chitosan are both hydrophilic[119]. Membranes containing these fillers have more equally distributed finger-like structures and fewer macro voids. The hydrophilicity of these fillers triggers a rapid non-solvent and solvent (NMP) interchange during the phase inversion activity, resulting in a change in membrane structure. Wider channels are created because of the quick exchange (finger-like structures). In the sphere of the circumstance of P membrane, the functional layer is on top although there is furthermore an irregular section underneath, portraying the polyethylene/polypropylene fabric that stayed as a support. Membranes in conjunction with P/CH/CN composite show better channel formation [120]

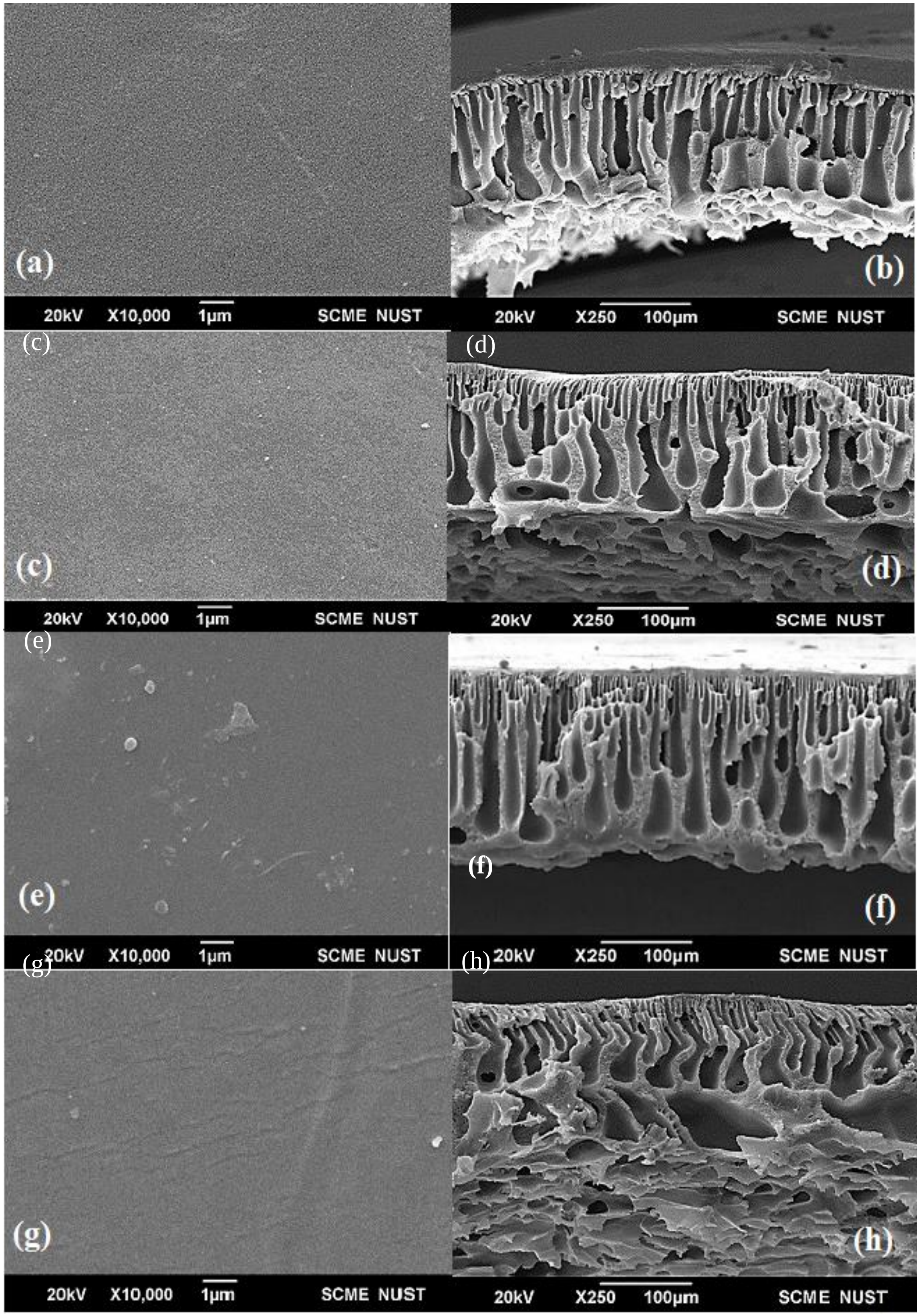


Figure 4.2 Topographic SEM images for (a) P, (c) PCH, (e) PCN, and (g) P/CH/CN and Cross-sectional SEM images for (b) P, (d) PCH, (f) PCN, and (h) P/CH/CN

4.3 Water Contact Angle

The addition of CNTs to the PES complex changed existing chemical characteristics of membranes, causing them to become more hydrophilic. The contact angle dropped from 74.74° to 61.26 ° in Fig. 4.3.

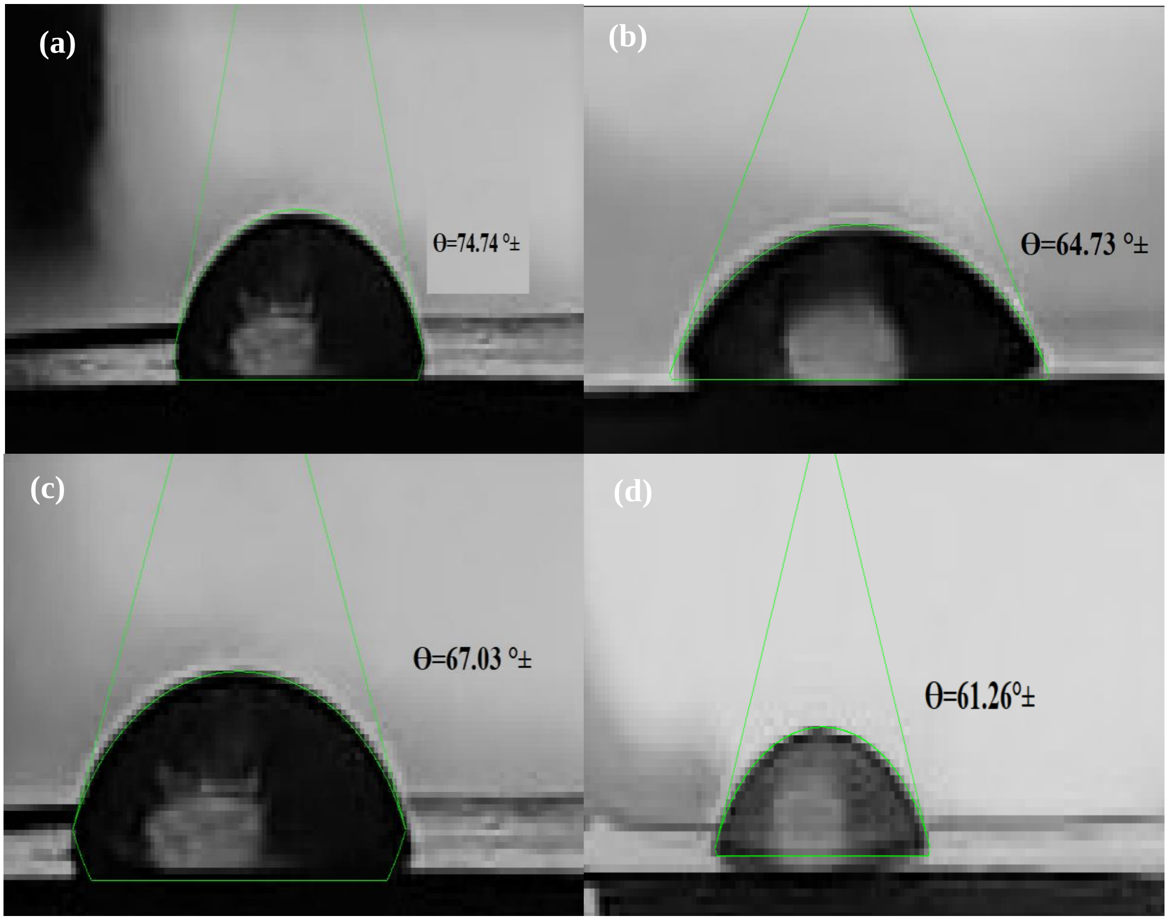


Figure 4.3 Average contact Angles for (a)P, (b)PCH, (c)PCN, (d)PCHCN

It reveals that incorporating Chitosan and Chitosan/CNTs complexes into the PES matrix considerably boosted the membranes' hydrophilicity, but a rise in the contact angle of PCN proposes the presence of CNTs on the surface, as CNTs are hydrophobic in nature [120] whereas P/CH/CN showed enhanced hydrophilicity. The contact angle measurement outcomes reach agreement with the roughness data from the AFM analysis [123–125].

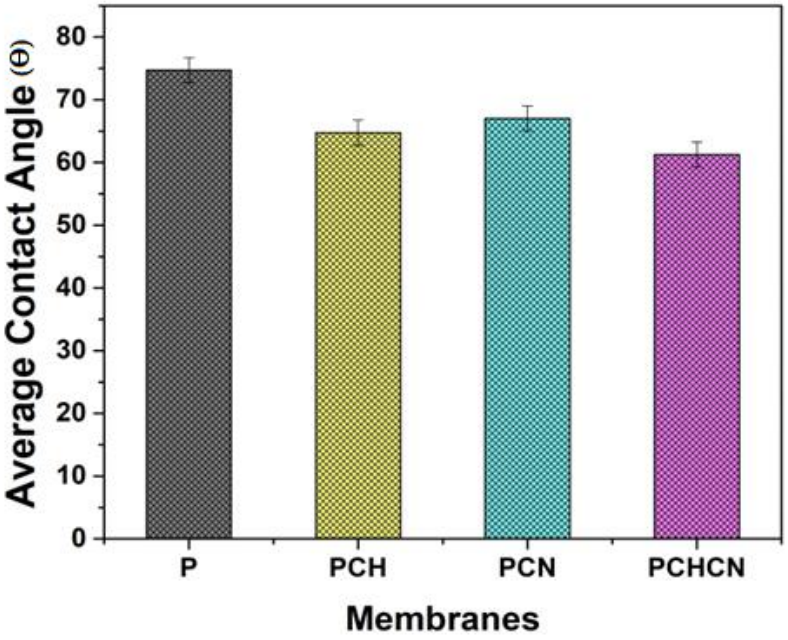


Figure 4.4 Contact Angle Bar Graph for Membranes

Table 4.1 Average contact Angles for Membranes

Membrane	**Average Contact Angle (Θ)**
P	74.74°±1
PCH	64.731°±1
PCN	67.03°±1
PCHCN	61.26°±1

4.4 Swelling ratio

Because of the hydrophobic nature of PES, the proportion for water retention was smallest in P, as shown in Figure 4.5. The asymmetric top layers of pure PES membranes were similarly less dense than the fillers in SEM micrographs, which might explain the lowered water uptake. The membrane P/CH/CN has the best water retention performance, with 91.2 percent.

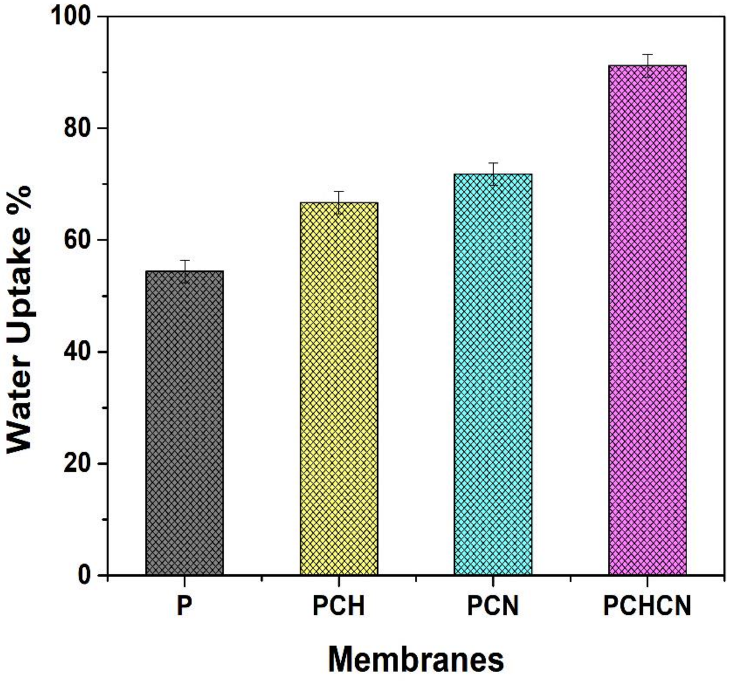


Figure 4.5 Water Uptake Bar Graph for Membranes

Table 4.2 Water Uptake of membranes

Membranes	Water Uptake %
P	54.39±1
P/CH	71.8±1
P/CN	66.7±1
P/CH/CN	91.2±1

The findings suggest that as the hydrophilicity of membranes increases, so does their ability to retain water[126] .Figure 4.5 shows that the composite membrane P/CH/CN outperformed the other membranes significantly. CNTs with chitosan were added and had a favorable effect. CNTs has a highly porous structure despite being hydrophobic. Water retention has improved because of the enhanced porosity [120, 127].

4.5 Porosity

A membrane's porosity is an essential factor. It affects the membranes' permeability, adsorption, and anti-fouling capabilities. For a good penetration flux, the membranes should be porous enough. The porosity of membranes can be influenced by hydrophilic fillers [128].The prepared P/CH/CN composite membrane had a high porosity of 77 percent, compared to 42.9 percent for the P membrane prepared without any filler[120, 129].

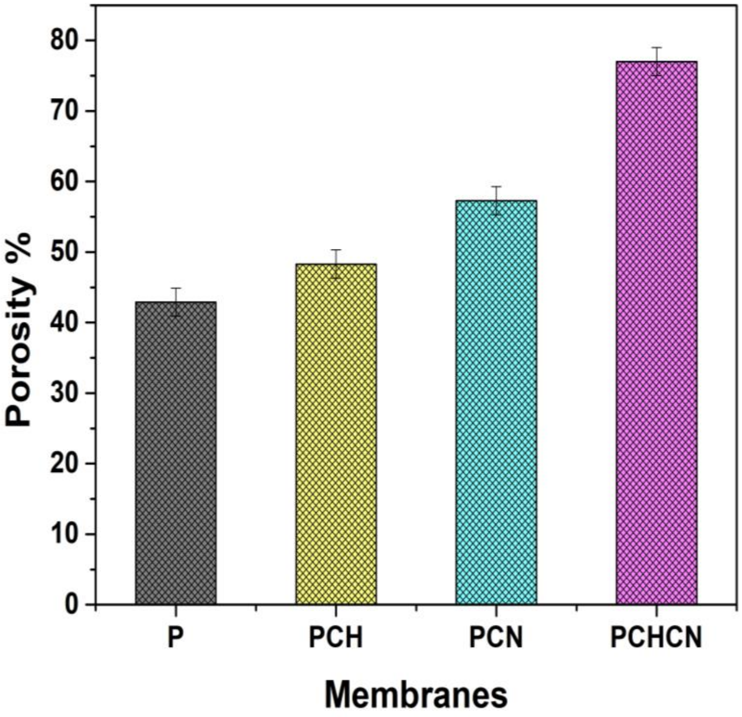


Figure 4.6 Percentage Porosity Bar graph for Membranes

4.6 Mechanical Testing

Membranes are used for a variety of purposes. Where the synthesis of multifunctional membranes is so common, membranes with strong mechanical strength are considered essential. Membranes with high polymer content have fewer voids and faults in their structure, which reduces the number of cracking sites [120]. Figure 4.7 shows the mechanical tensile strength curves.

P membranes showed more strength than PCH this is owing to the solubility of chitosan, as its soluble in water [120, 130, 131]. By means of water-soluble fillers possibly will outcome as lower mechanical qualities, when these soluble molecules have a tendency to enhance macro void formation during immersion, compromising the membranes' mechanical strength. Water dissolves materials with a higher water solubility. As a result, voids appear where the material previously existed. These spaces then serve as new places for the fracture to spread, leading to membrane tear [120]

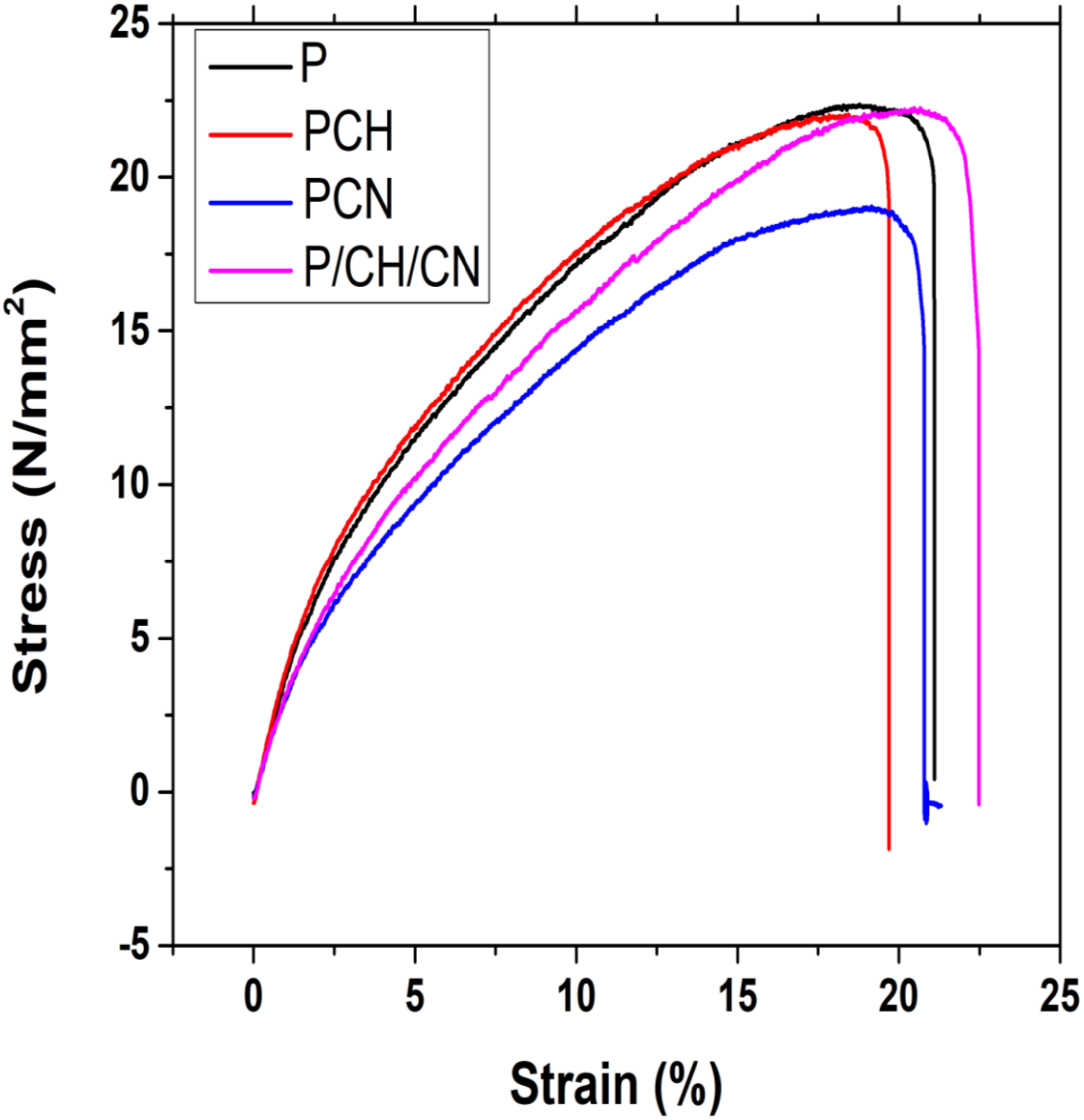


Figure 4.7 Stack Graphs for Mechanical Properties

In PCN, inadequate distribution and accretion of CNTs in the polymer matrix resulted in a decrease in membrane mechanical strength and modulus. Interfacial interactions between MWCNTs and polymer matrices also have a substantial impact on the mechanical properties of the composites[132]. It has also been reported in literature that CNTs concentrations up to 0.1% show a decrease in mechanical strength of membrane [133].

The highly dispersed CNTs/Chitosan composite interlaced inside the polymer matrix operate as links, increasing interfacial interactions across polymeric chains and those between the CNTs/Chitosan composite and the polymer matrix, resulting in maximal strength for P/CH/CN. The nanofiller must meet four major criteria to effectively strengthen the polymer composite: (a) large aspect ratio; (b) excellent dispersion; (c) alignment; and (d) interfacial stress transmission. The objective of carbon nanotubes in composites is that when external stress is applied to the composite, a portion of that stress can be transferred to the CNTs, allowing the nanoparticles to take on more of the load [134].

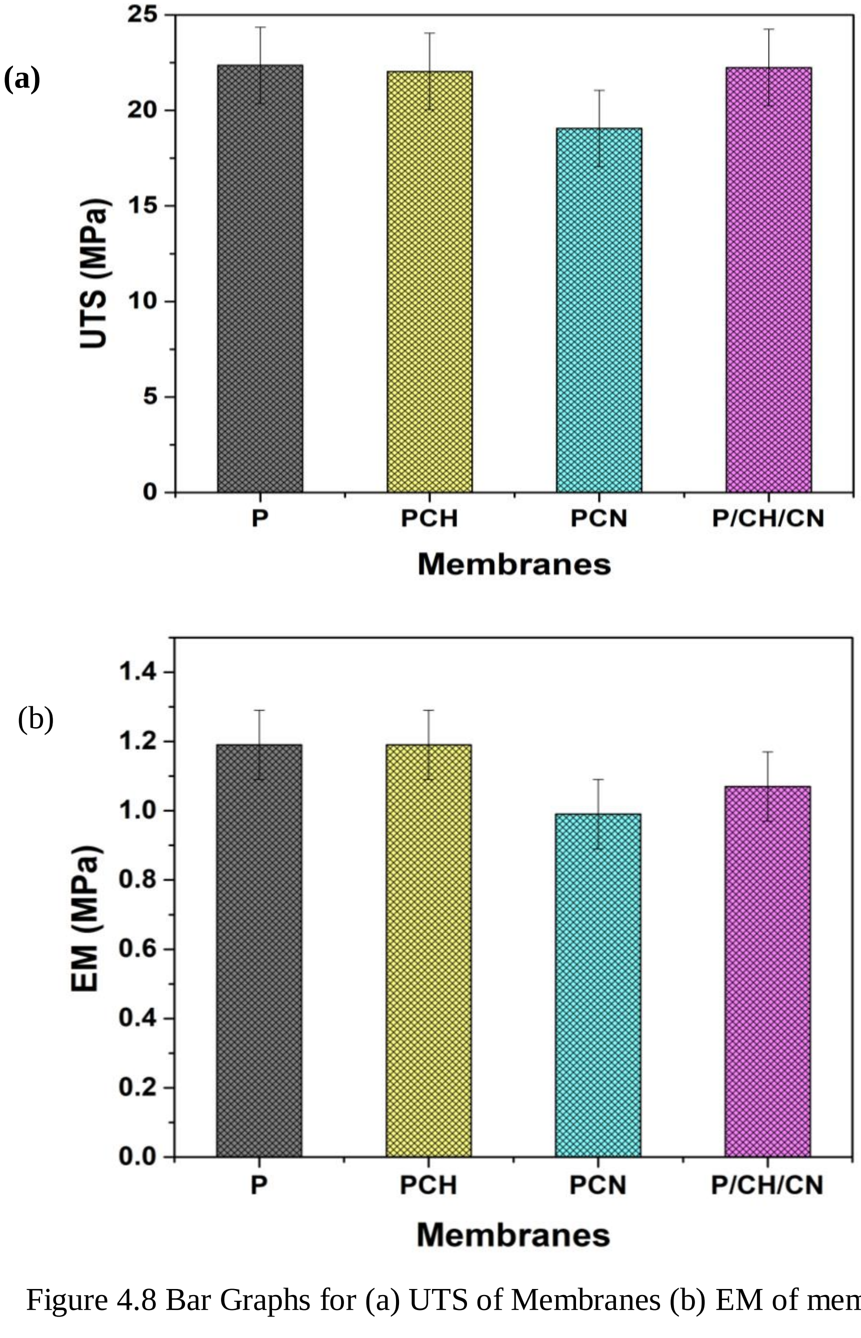


Figure 4.8 Bar Graphs for (a) UTS of Membranes (b) EM of membranes

4.7 AFM

Using atomic force microscopy, the impacts of chitosan and CNT inclusion on membrane surface roughness were investigated (AFM). The average arithmetic roughness (Ra) values decreased for PCH with the addition of chitosan before rising for PCN.

This initial decrease in membrane surface roughness may be attributed to the casting solution's increased viscosity after the addition of Chitosan, which slows the rate during which solvent and non-solvent interchange, resulting in a smoother membrane surface [135].

Additionally, as seen in PCH and PCHCN, the decline in membrane surface roughness is a result of the development of reduced surface pores, as shown by SEM analysis. Due to stronger van der Waals force, CNTs bundle and aggregate under high loadings, creating a rougher membrane surface, like PCN [136–138].

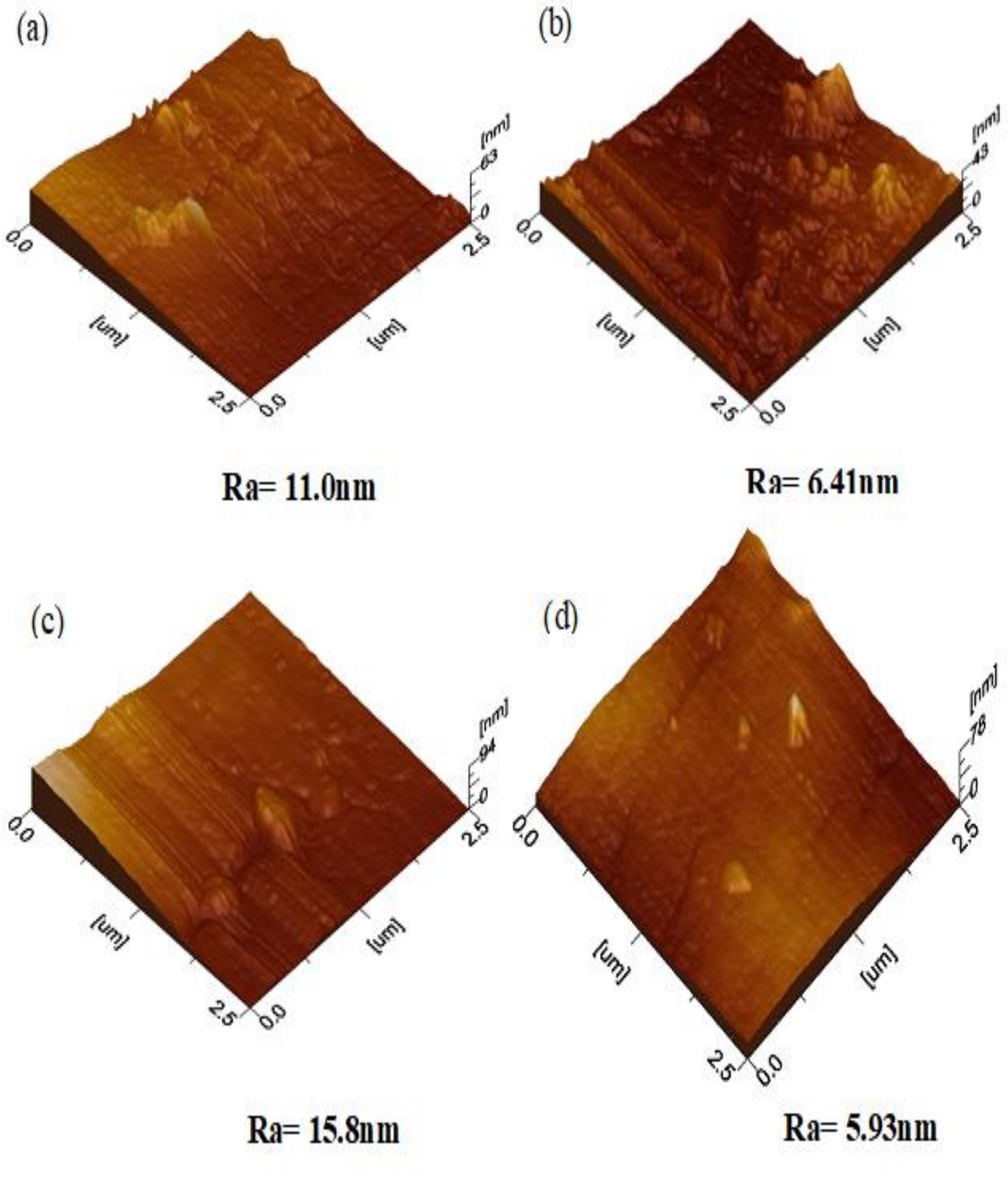


Figure 4.9 AFM Roughness images of membranes(a) P, (b)PCH, (c)PCN, (d)PCHCN

4.8 Flux

The response surface analysis for the membrane permeability of P, PCH PCN, and P/CH/CN for the model is shown in Figures 4.10-4.11. Increases in feed temperature and pressure increase permeate flux in all conditions for all membranes, according to the response surface. Figure 4.10 shows the highest flow rate obtained from the model for P at 1500 mg/L feed concentration and 6 kg/cm^2 feed pressure.

The model also found a similar tendency for the other membranes. P membranes have a significantly higher permeate flux than other membranes. This is because the permeate flow is restricted by the membrane pore size. P, PCH, PCN, and P/CH/CN had the greatest fluxes of 841.2, 568.2, 447.2, and 436.8 Lm^{2}h^{-1} respectively.

The lowest fluxes were 200.4, 202.8, 115.91, and 103.8 Lm^{2}h^{-1} for P, PCH, PCN, and P/CH/CN, respectively. Figure 4.14 shows Predicted vs experimental (actual) data points for the determination of variance in the flux of membrane samples.

4.9 Salt Rejection

The response plot for salt rejection of synthesized membranes is shown in Figures 4.12-4.13. The findings reveal that as feed temperature rises, salt rejection rises as well. With a decrease in temperature and a rise in pressure from 4 to 6 kg/cm, salt rejection increases significantly for high and low feed concentrations.

The highest salt rejection values were 62.3, 83.7, 92, and 95.34 percent for P, PCH, PCN, and P/CH/CN, respectively. The lowest values of salt rejection recorded for P, PCH, PCN, and P/CH/CN were 52.8, 73, 86.5 and 91% respectively.

Such remarkable removals have also been reported in the past [114, 116, 139]The results are explained in Table 4.4. Figure 4.15 shows predicted vs actual values of % salt rejection of membrane samples.

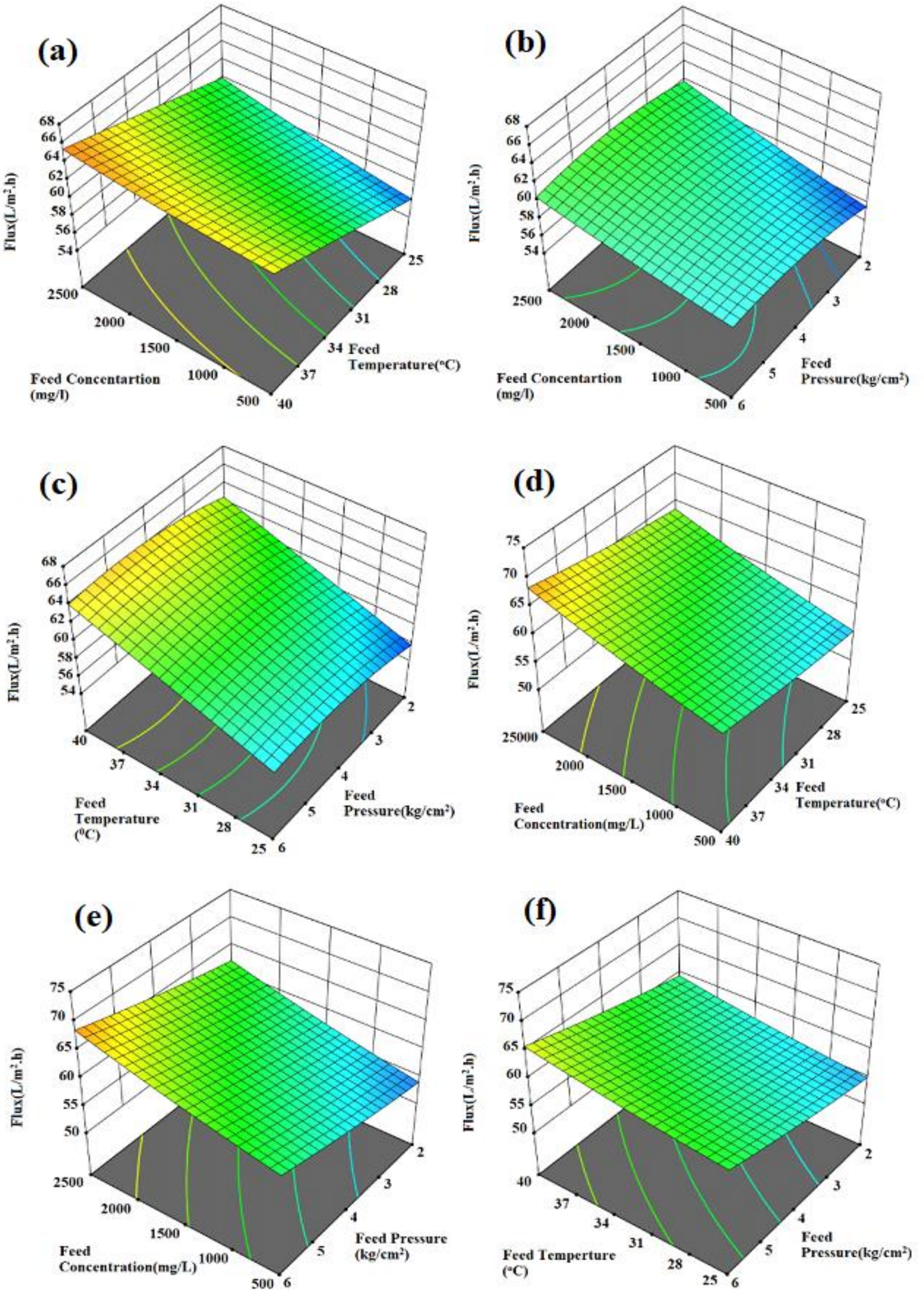


Figure 4.10 Effect of feed concentration, temperature, and pressure on the flux through (a-c) P and (d-f) PCH membrane samples.

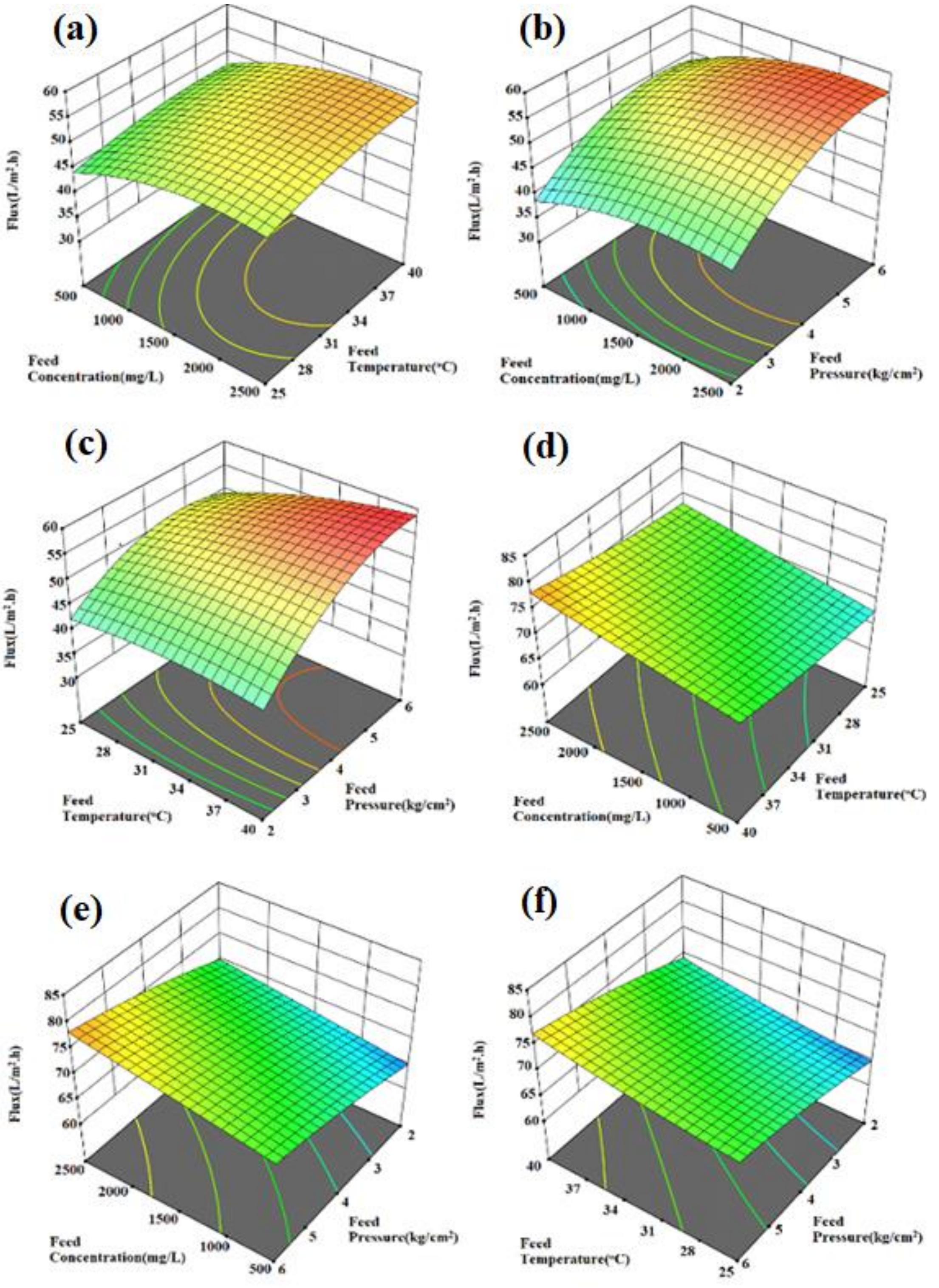


Figure 4.11. Effect of feed concentration, temperature, and pressure on the flux through (a-c) PCN, and (d-f) PCHCN membrane samples

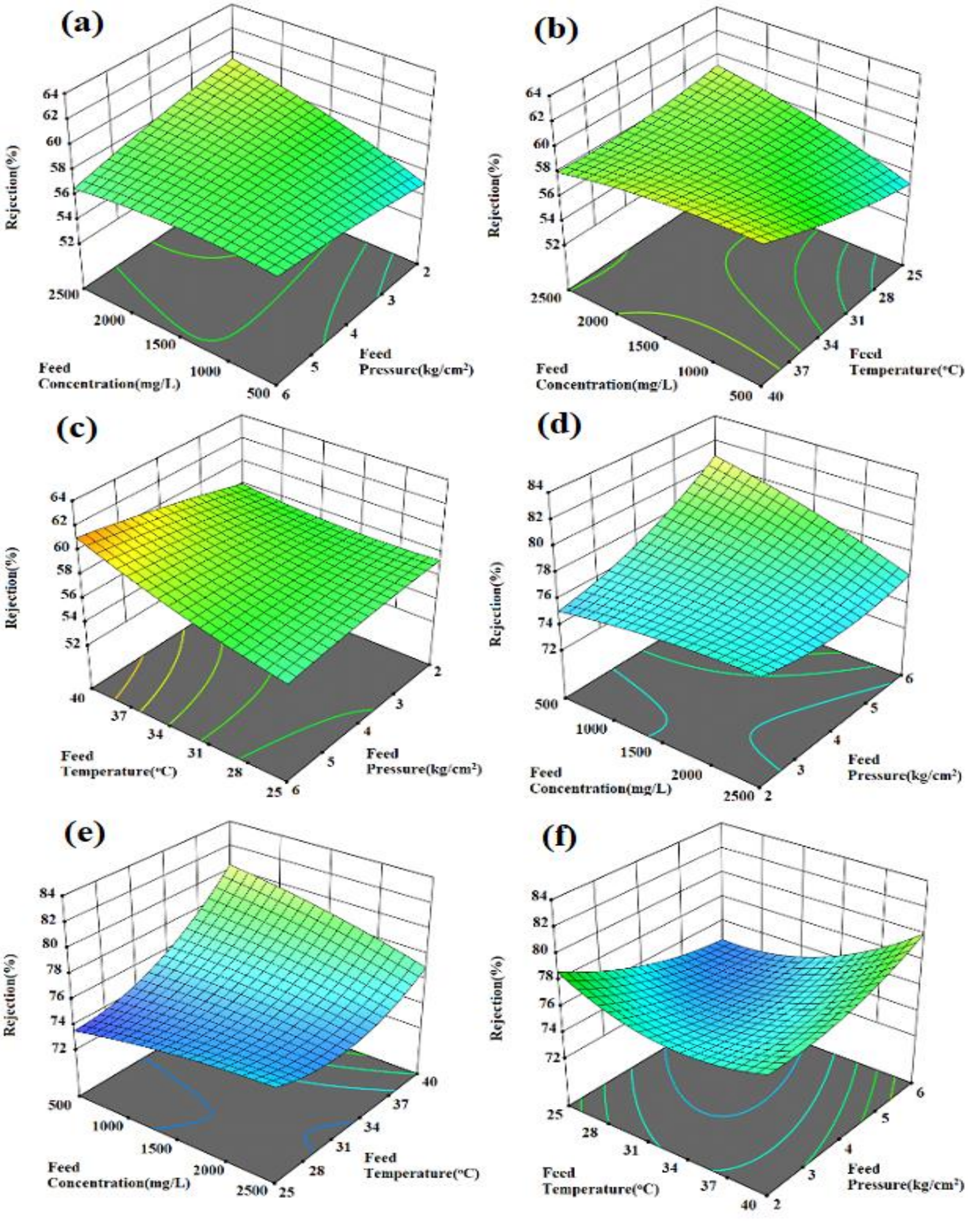


Figure 4.12. Effect of salt concentration in the feed water, temperature, and pressure of the feed water on the % salt rejection by the (a-c) P, and (d-f) PCH membrane samples.

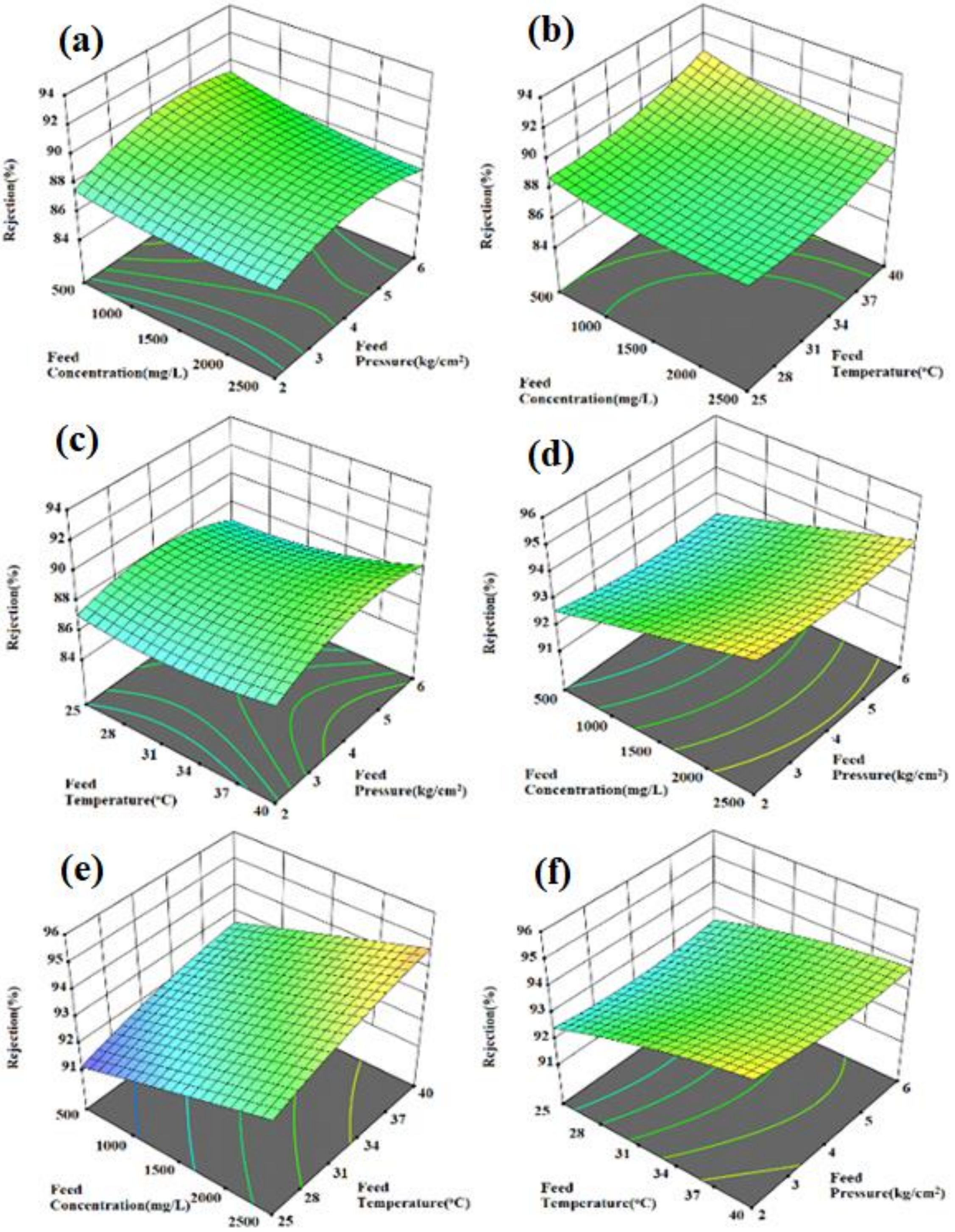


Figure 4.13 Effect of salt concentration in the feed water, temperature, and pressure of the feed water on the % salt rejection by the (a-c) PCN, and (d-f) PCHCN membrane samples.

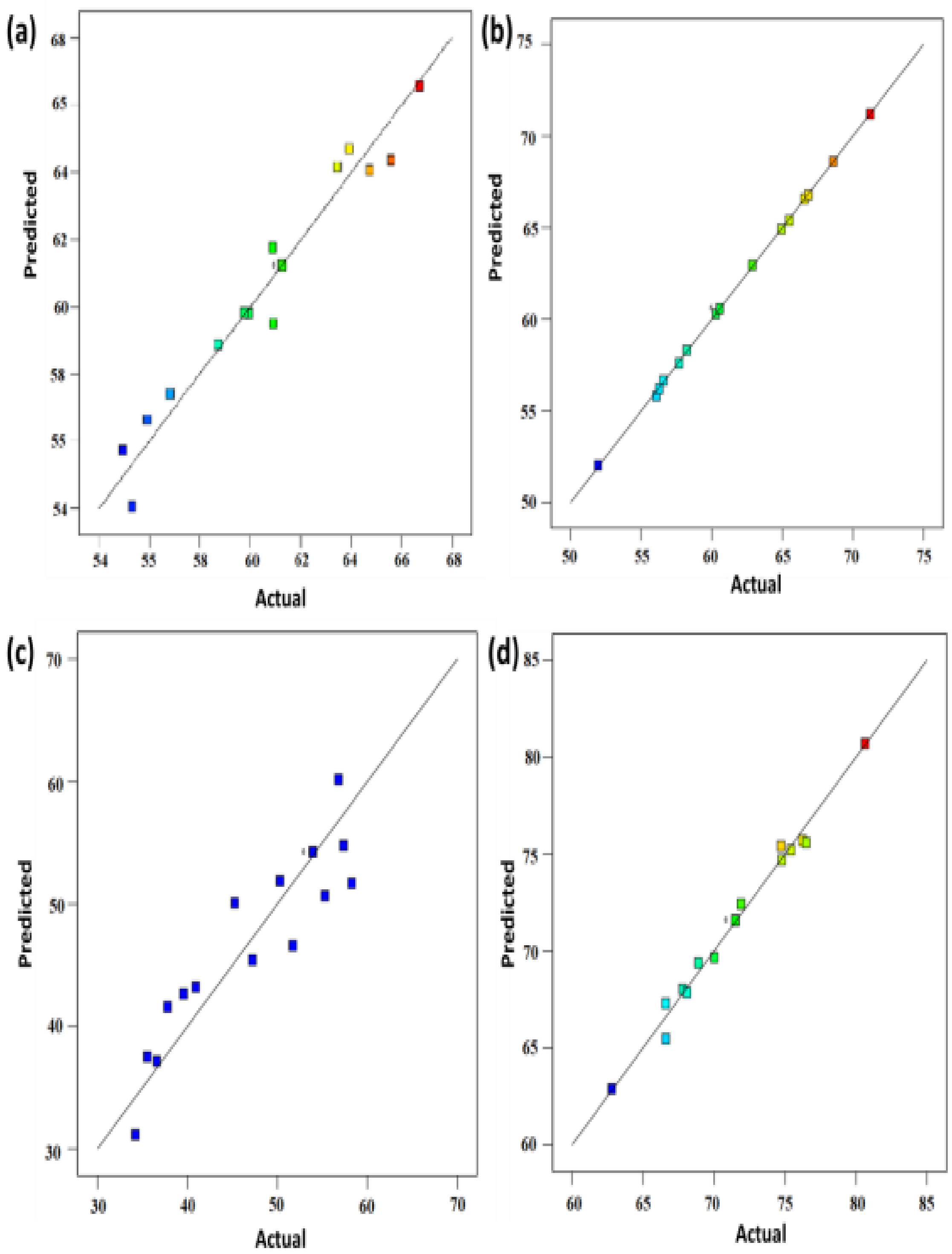


Figure 4.14 Predicted vs experimental (actual) data points for the determination of variance in the flux of (a) P, (b) PCH, (c) PCN, and (d) PCHCN membrane samples

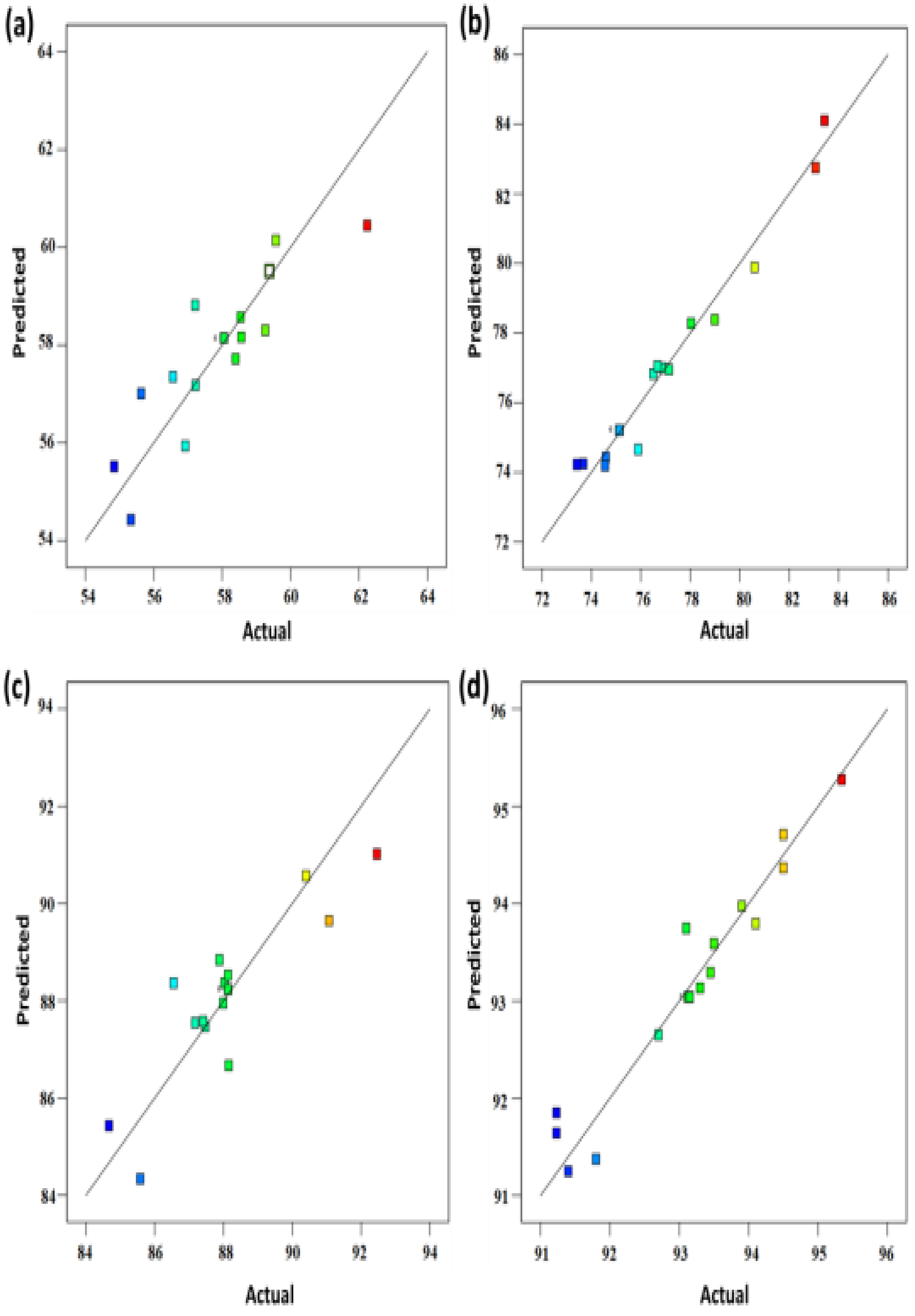


Figure 4.15 Predicted vs actual values of % salt rejection from (a) P, (b) PCH, (c)PCN, and (d)PCHCN membrane samples

Table 4.3 Experimental Design and responses of Membranes

Runs	**Factors**			**Responses**							
	Factor: A	**Factor: B**	**Factor: C**	**R_1**				**R_2**			
				P	**PCH**	**PCN**	**PCHCN**	**P**	**PCH**	**PCN**	**PCHCN**
1	1500	32.5	0.636414	209.2	214.4	250.6	102	55.22	77.14	85.58	93.1
2	3181.79	32.5	4	290.8	481.7	137.4	129	54.56	73.41	88.13	91.23
3	1500	32.5	7.36359	252.8	558.2	447.2	207	60.2	78.03	87.17	93.12
4	1500	32.5	4	841.2	400.8	259.8	234	57.8	75.13	88.13	91.23
5	1500	32.5	4	841.2	400.8	478.98	436.8	57.8	75.13	87.41	94.5
6	500	25	2	579.6	277.2	259.8	285.6	55.3	73.67	92.46	92.7
7	1500	45.1134	4	413.8	515.3	259.8	207	62.2	83.07	86.55	93.15
8	2500	40	6	452.4	407.4	251.2	207	57.2	78.99	91.07	93.15
9	1500	32.5	4	841.2	400.8	259.8	236.5	57.8	75.13	88.13	95.34
10	2500	25	6	352.4	387.6	259.8	224	58.3	74.55	87.99	94.5
11	1500	32.5	4	841.2	400.8	415.3	158.7	57.8	75.13	90.4	94.1
12	181.793	32.5	4	790	209.8	358.7	207	61.9	75.89	88.13	93.15
13	500	40	6	354.6	568.2	451.2	225	60.5	83.43	88.13	93.45
14	500	40	2	612	568.2	259.8	207	55.6	76.5	87.88	93.15
15	1500	32.5	4	841.2	400.8	258.3	207	57.8	75.13	84.67	93.15
16	1500	19.8866	4	200.9	200.4	340.4	103.8	56.56	76.67	88.13	93.3
17	1500	32.5	4	841.2	400.8	115.91	114	57.8	75.13	88.15	91.8
18	500	25	6	606	505.2	311.2	342	52.8	74.58	87.47	93.9
19	2500	40	2	252.8	158.4	130.21	198.5	60.5	76.88	88.13	93.5
20	2500	25	2	202.8	202.8	204.7	253.4	62.3	80.6	88.03	91.4

R_1: Flux of membranes, R_2: Salt Rejection of membranes

Table 4.4 Summary of properties for fabricated membranes

Properties	**P**	**PCH**	**PCN**	**PCHCN**
Water retention (%)	54.39±1	71.8±1	66.7±1	91.2±1
Gravimetric analysis (%)	42.9±1	57.3±1	48.3±1	77±1
Contact Angle (θ)	74.74°±1	64.731°±1	67.03°±1	61.26°±1
Surface Roughness (nm)	11.0±0.1	6.41±0.1	15.8±0.1	5.93±0.1
Young Modulus (MPa)	1.19±0.1	1.19±0.1	0.99±0.1	1.07±0.1
Ultimate Tensile Strength (MPa)	22.2436±0.1	22.0405±0.1	19.0558±0.1	22.3584±0.1

Conclusion

Polymer CNT/chitosan nanocomposite membranes were prepared through phase inversion method. The additives were Polyvinylpyrrolidone (PVP), Chitosan, and Carbon nanotubes (CNTs). ATR-FTIR results confirm the functional groups for PES. Response surface methodology was used to investigate the effect of feed temperature, feed concentration and feed pressure on flux and salt rejection. The salt rejection rates for the polymer CNT/chitosan were remarkable for Na_2SO_4 salt removal. The contact angle result also demonstrates that the CNT/chitosan composite membrane has higher hydrophilic properties. With the addition of composite, porosity and water uptake abilities improved dramatically. The mechanical strength of membrane containing chitosan, which is soluble in water, diminishes, resulting in the creation of micro spaces and a reduction in mechanical strength. These findings reveal that CNT/chitosan composites have considerable potential for water desalination and should be investigated further. Response Surface Methodology model showed a complete fit for these membranes.

Recommendations

On the account of the presented study its recommended to use this novel composite to fabricate this novel membrane for adsorption of heavy metals and dye removal from water. Further modification by functionalization in the membrane fabrication is recommended to achieve desalination for other salts.

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