SYNTHESIS, CHARACTERIZATION AND APPLICATION OF POLYMER NANOCOMPOSITE MEMBRANE

ARJYAMA BORUAH
ADITYA BARKATAKY
RAKTIM JYOTI DEKA
RAJPRATIM MEDHI

DEPARTMENT OF CHEMICAL ENGINEERING
ASSAM ENGINEERING COLLEGE
GUWAHATI-781013
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OF
POLYMER NANOCOMPOSITE MEMBRANE

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ARJYAMA BORUAH (20/009)
ADITYA BARKATAKY (20/122)
RAKTIM JYOTI DEKA (20/188)
RAJPRATIM MEDHI (20/411)

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This is to certify that Arjyama Boruah (20/009), Aditya Barkataky (20/122), Raktim Jyoti Deka (20/188) and Rajpratim Medhi (20/411) of B.Tech 8th Semester have jointly carried out the project entitled “**SYNTHESIS, CHARACTERIZATION AND APPLICATION OF POLYMER NANOCOMPOSITE MEMBRANE**” under my supervision and submitted the report in fulfilment of the requirement for the Degree of Bachelor of Technology in Chemical Engineering of Assam Science and Technology University, which may be accepted.

Date:

Dr. Bandana Chakrabarty Head Of Department, Department of Chemical Engineering Assam Engineering College	Mr. Souradip Choudhury Assistant Professor Department of Chemical Engineering Assam Engineering College

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ABSTRACT

This research contributes to the understanding of Polymer Nano-composite membranes and their potential in removing aqueous contaminants. The objective of our project was to synthesize and characterize a Polymeric Nanocomposite Membrane, for application in the removal of Industrial wastewater Effluents, especially Methylene Blue. For our experiment, Polysulphone was taken as the Polymer, DMAC (Dimethyl acetamide) as the solvent and TiO_2 / GO as the nanomaterial, while the membranes were synthesized using the Phase Inversion Technique.

Several characterization techniques were carried out, including FESEM to study the surface morphology and thickness of the membrane cross section, contact angle to determine the membrane hydrophilicity, Batch Cell Setup to determine the flux through the membranes, which is later used to find the average pore size of the membranes among others.

The main objective of incorporating nanomaterials was to reduce the hydrophobicity of the membranes, which was supported by experimental results from the contact angle experiment. However, it was observed that increasing the content of Nanomaterials lead to a reduction in the porosity percentage of the membranes.

Additionally, it was observed that the pore size of the membranes decreases with the addition of nanomaterials, which was obtained from the Batch cell experimental setup with the help of Darcy’s Law and Hagen-Poiseuille’s equation. Thus, we infer that membranes incorporated with Nanomaterials will be better efficient in removal of finer particles, especially those in the range of Ultrafiltration.

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LIST OF NOMENCLATURES USED

S.no	Property	Symbol
1	Permeability	k
2	Membrane thickness	t
3	Porosity	Ɛ
4	Viscosity	μ
5	Specific heat	C_P
6	Tortuosity	τ
7	Pore size diameter	d_p

CHAPTER 01

INTRODUCTION

1.1 INTRODUCTION TO MEMBRANES

A membrane is a thin, flexible layers of material that act as selective barriers, allowing certain substances to pass through while blocking others, depending on various criteria such as size, shape, or chemical properties. Membranes are integral to a wide array of applications, including water treatment, gas separation, and medical sector, by enabling efficient processes for filtration, separation, and purification. Their ability to provide precise control over molecular and ionic movement makes them vital components in modern technological studies. By facilitating the selective transport of materials, membranes help achieve high efficiency and effectiveness in numerous industrial and technological processes, underscoring their vital role in advancing modern engineering solutions, hence a key topic of study in today's world.

1.2 MEMBRANE SEPARATION AND OTHER SEPARATION METHODS

Membrane separation is a process that utilizes the micropores of the membranes and their selective permeability to separate components of a feed mixture based on their size, shape, charge, or other properties. Membrane separation methods include microfiltration, ultrafiltration, nanofiltration, and reverse osmosis, each tailored to specific separation requirements.

TABLE 01: Different available methods for separation

Separation Technique	Disadvantages
Distillation: Separates components in a liquid mixture based on differences in boiling points, or volatility difference	• High energy consumption • Requires the components to have a considerable volatility difference • Limited to liquid mixtures

Extraction: Separates components of a feed on the basis on differences in solubility.	• Energy Intensive • Requires the usage of a solvent that has high miscibility with the solute. • Expensive solvent regeneration
Absorption: Separates solutes from a Gas stream by absorbing the solute into the Liquid Bulk.	• Limited Tower Capacity • Discharge of solute rich untreated Liquid Streams • High energy requirements • Applicable only to Gas-Liquid systems
Chromatography: Separates components based on differences in partitioning behaviour between a stationary phase and a mobile phase.	• Time consuming • High Cost equipment required • Limited scalability

1.2.1 ADVANTAGES OF USING MEMBRANE SEPARATION:

1. **Energy Efficiency**: Membrane separation processes typically require lower energy inputs compared to thermal separation methods like distillation.

2. **Selective Separation**: Membranes offer precise control over separation, allowing for selective removal or concentration of specific components.

3. **Minimal Chemical Usage**: Membrane separation requires minimal chemicals, making it environmentally friendly and reducing operational costs.

4. **Compact Design**: Membrane systems often have a smaller footprint compared to other separation equipment, making them suitable for space-constrained environments.

5. **Versatility**: Membrane materials and configurations can be tailored to suit a wide range of separation needs, offering flexibility in application across diverse industries.

Overall, membrane separation offers a cost-effective, efficient, and environmentally friendly solution for various separation challenges, making it a preferred choice in many industrial processes.

1.3 CLASSIFICATION OF MEMBRANES

Membranes are broadly classified into 2 categories based on their composition:

- Organic Membranes
  Ex: Polymeric Membranes, Cellulosic Membranes

- Inorganic Membranes
  Ex: Ceramic Membranes, Metal Membranes

1.3.1 ORGANIC MEMBRANES

Organic membranes are materials primarily composed of carbon-based (organic) compounds that are used for separation processes, filtration, and other applications. These membranes leverage the unique properties of organic materials to achieve specific functionalities. They control the passage of different substances, such as gases, liquids, ions, or particles, based on specific criteria like size, charge, or chemical affinity.

TABLE 02: Different types of Organic Membranes

Type of membrane	Composition	Characteristics	Applications
Cellulosic Membranes	Cellulose acetate Cellulose nitrate	➢ Bio-compatible ➢ Hydrophilic ➢ Lower Cost	➢ Dialysis ➢ Microfiltration ➢ Water treatment
Polymeric Membranes	Polysulfone based Polyamide based Polyvinyl based	➢ Chemical resistance ➢ Mechanical strength	➢ RO and NF in water purification ➢ Gas separation applications
Biodegradable Polymer Membranes	Polylactic Acid, Polycaprolactone	➢ Biodegradable ➢ Environment-safe	➢ Tissue engineering ➢ Drug delivery ➢ Bio-packaging
Polymer Nanocomposite Membranes	Polymer matrix blended along with nanoscale fillers or nano-additives	➢ High Mechanical Strength ➢ Enhanced Selectivity ➢ Highly Tailorable properties	➢ UF Water purification ➢ Gas separation ➢ Biomedical applications

1.3.2 INORGANIC MEMBRANES

Inorganic membranes are membranes made from inorganic materials such as ceramics, metals, and semiconductors, as opposed to organic membranes made from polymers. These membranes are typically used for various separation processes, including gas separation, liquid filtration, and ion exchange. Inorganic membranes offer certain advantages over organic membranes, including higher chemical and thermal stability. However, Inorganic membranes are typically more expensive to produce and may have more limited flexibility and scalability compared to organic membranes. They find applications in industries such as water treatment, pharmaceuticals, food and beverage processing, and energy production.

TABLE 03: Different types of Inorganic Membranes

Type of membrane	Composition	Characteristics	Applications
Ceramic Membranes	Alumina, silica, zirconia, titania, etc.	➢ Thermal Stability ➢ Chemical Stabiluty ➢ Mechanical strength	➢ Gas separation ➢ Catalysis ➢ Water treatment
Metal Membranes	Stainless steel, palladium, nickel, etc.	➢ Resistance to high temperatures ➢ High Mechanical strength ➢ Resistant to Corrosion	➢ Hydrogen separation ➢ Gas purification ➢ Catalytic reactions
Zeolite Membranes	Crystalline aluminosilicate materials	➢ Uniform Pore Structure ➢ Enhanced Selectivity	➢ Gas separation ➢ Ion exchange ➢ molecular sieving
Polymeric Inorganic Membranes	Inorganic matter combined with polymers	➢ Enhanced selectivity ➢ Enhanced Permeability ➢ High Mechanical Strength	➢ Application fields under research at present

1.4 FACTORS INFLUENCING THE SELECTION OF THE TYPE OF MEMBRANE TO BE SYNTHESIZED

Water is laden with numerous pollutants, encompassing toxic heavy metals, pesticides, dyes, organic acids, halogenated compounds, fertilisers, and microorganisms. Among these pollutants, heavy metals pose a particularly severe threat to ecosystems and organisms due to their non-biodegradable and toxic nature. These hazardous metals tend to accumulate in ecosystems, particularly within the food chain and living organisms. The intake of polluted water has far-reaching consequences on human health, manifesting in various problems such as organ damage, skin irritation, cancer, rupture of the nasal septum, diarrhoea, loss of appetite, abdominal pain, and headaches. Given the detrimental impact of these pollutants, their removal is imperative to ensure clean water for the well-being of all living creatures. In response to this challenge, advanced membrane technologies have emerged as effective solutions for the removal of contaminants from water and wastewater. These technologies play a crucial role in safeguarding the health of ecosystems and organisms, facilitating the provision of clean water for sustenance, and promoting a healthier life.

One such membrane is the polymeric membrane, a thin layer of material composed of a polymer matrix. These membranes are engineered to have specific properties that make them useful in various applications, particularly in separation processes. The unique characteristics of polymeric membranes make them valuable, especially for water treatment and allied fields.

1.4.1 BENEFITS FOR USING POLYMERIC MEMBRANES

- Polymeric membranes provide **Selective Permeability** (can be designed to selectively allow certain substances to pass through while blocking others based on size, charge, or other properties).

- Polymeric membranes are typically **lightweight**, which simplifies handling and installation. This is advantageous in applications where weight considerations are important, such as in portable water purification devices.

- Polymeric membrane separation processes are **Energy-Efficient**, compared to traditional separation techniques, as there is either no or minimal usage of power operated devices.

- Compared to other types of membranes, such as ceramic or metallic membranes, polymeric membranes are often **Cost-effective** to manufacture and install.

- Owing to the lower costs, water purification setups made from polymeric membranes are thus **Scalable,** and more suitable for research related studies.

- The Polymeric membranes offer higher degree of **Flexibility and Reusability** as compared to other membranes, which can be only used for one cycle and cannot be bent or shaped.

1.4.2 PROPERTIES ENHANCED BY THE ADDITION OF NANOMATERIALS

The major challenge in a polymeric membrane separation is fouling. Membrane fouling, where particles or substances accumulate on the membrane surface over time, can reduce efficiency, and require frequent maintenance. Along with fouling some polymeric materials may also degrade or lose their properties over time, affecting the performance and lifespan of the membrane.

To address this issue, the inclusion of nano-sized materials in the polymer matrix has made significant progress in overcoming the challenges of water treatment of polymeric membranes developed and synthesised. In addition, polymeric membranes infused with nanomaterials have pore size in the range similar to the water based contaminants, thereby increasing their removal efficiency. Thus, Nano-based polymeric membranes represent an advanced class of Polymeric membranes that incorporate nanomaterials or nanotechnology to enhance their properties and performance.

Advantages of incorporating nanomaterials into Polymeric membranes are discussed as below:

- **Anti-fouling Properties:** Nanomaterials enhance the anti-fouling characteristics of polymeric membranes, such as hydrophilic coatings or functionalized nanoparticles, help prevent the accumulation of contaminants on the membrane surface, thereby reducing fouling.

- **Increased Thermal Stability**: Incorporating thermally stable nanomaterials into the polymer matrix improves the membrane's overall thermal resistance, enabling use in higher-temperature environments.

- **Improved Chemical Resistance:** The incorporation of chemically resistant nanoparticles or nanocomposites enhances the membrane's resistance to harsh chemicals, expanding its applicability in various chemical processes.

- **Improved Mechanical Strength:** The addition of nanomaterials, reinforces the polymeric matrix, enhancing the mechanical strength, rigidity and durability of the membrane.

- **Tuning Surface Properties:** Nanomaterials allow for surface modification, enabling the tuning of surface properties such as hydrophilicity or hydrophobicity to meet specific application requirements.

- **Reduction in the membrane pore diameter:** Addition of nanomaterials into the Polymer membrane matrix leads to the reduction in the membrane Pore size, thereby making the membrane capable of filtering finer particles, in the range of Ultrafiltration, which is similar to the size of the water-contaminants that are targeted to be removed.

TABLE 04: Effect of Pore size on Particle Separation

FILTRATION TYPE	PARTICLE RANGE	APPLICATION
Micro-Filtration (MF)	100-10,000 nm	Used for separation of larger particles from liquids bulks/streams
Ultra-Filtration (UF)	10-100 nm	Suitable for separation of Water-contaminants like Dyes like Methylene Blue, Oil-Water Emulsions, etc
Nano-Filtration (NF)	1-10 nm	Applications in the separation of ions and small molecules based on size and charge
Reverse Osmosis (RO)	0.1-1 nm	Effective for Desalination and removal of ultrafine solutes from water and other liquids

1.5 FABRICATION TECHNIQUES USED TO SYNTHESIZE POLYMERIC NANOCOMPOSITE MEMBRANES

1.5.1 PHASE INVERSION PROCESS

The phase inversion process for manufacturing poly-nanocomposite membranes involves transforming a homogeneous polymer solution into a solid membrane through thermodynamic changes, typically induced by solvent removal. This summary outlines the steps:

- Polymer Solution Preparation: Dissolve the Polymer in appropriate solvent selected depending on the Polymer-Solvent Combination.
- Nanofiller Dispersion: Disperse the nanoparticles into the polymer solution, ensuring uniform distribution using magnetic stirring.
- Solution Casting: Apply the polymer-nanofiller solution onto a substrate via methods such as doctor-blading, influencing membrane structure and properties.

- Solvent Evaporation: Allow the solvent to evaporate through air drying or controlled heating, inducing phase separation into polymer-rich and solvent-rich phases.
- Quenching/Coagulation: Immerse the membrane in a coagulation bath to enhance phase separation and solidification, affecting morphology and pore size.
- Rinsing and Drying: Rinse the membrane to remove residual solvent, then immerse it in deionized water for 12-24 hours. Finally, dry the membrane to obtain the finished poly-nanocomposite membrane.
- This immersion precipitation technique ensures the creation of a solid membrane with desired properties through controlled phase separation and solvent removal.

1.5.2 INTERFICIAL POLYMERIZATION PROCESS

The Interfacial Polymerization method involves the reaction of two or more monomers at the interface between two immiscible phases, typically a liquid-liquid or a liquid-gas interface. The reaction occurs at the interface, leading to the formation of a thin polymer film. When nanoparticles are incorporated into this process, it results in the formation of poly nanocomposite membranes with enhanced properties.

It is a widely favoured method for producing Polymeric Nanocomposite Membranes (PNCMs), especially for critical applications like nanofiltration (NF), thin-film composite (TFC), and reverse osmosis (RO). IP's significance lies in creating TFC membranes that have exceptional properties, including a microporous substrate layer and customizable skin layer optimization. This technique has also been effectively employed for producing RO and NF membranes, both having diverse applications.

Following gives a general overview of the Interfacial Polymerization technique for preparing poly nanocomposite membranes:

- Two different monomers are selected, each dissolved in a separate phase. Commonly, an aqueous phase and an organic phase are used, and the monomers should be reactive with each other.
- The two phases are brought into contact to form an interface. This interface can be either liquid-liquid or liquid-gas, depending on the specific requirements of the synthesis.

- Initiators or catalysts are added to induce the polymerization reaction. The monomers diffuse to the interface and react with each other at the interface, leading to the formation of a polymeric film.
- Nanoparticles are introduced into one or both of the monomer phases. These nanoparticles can be dispersed throughout the polymer matrix during the polymerization process.

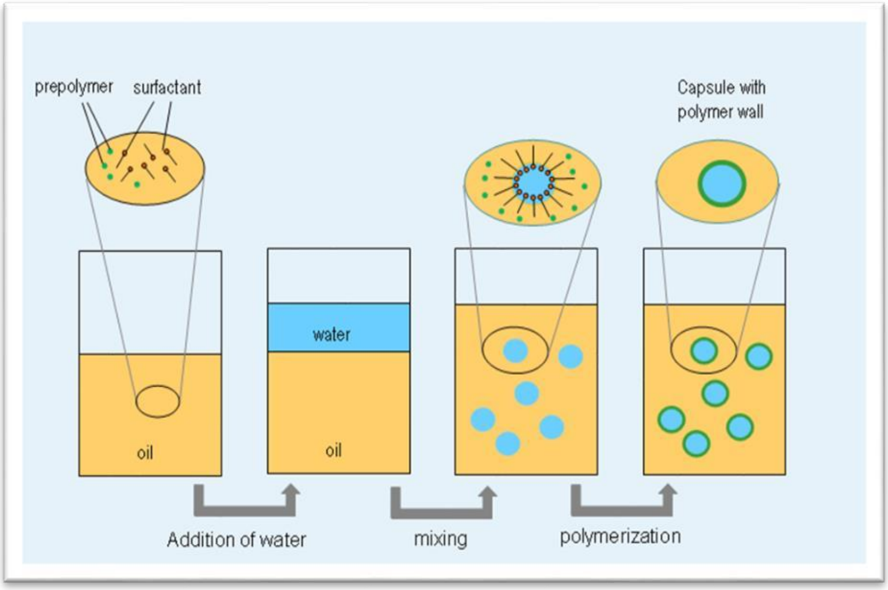


Fig 01: Schematic diagram of interfacial polymerization

1.5.3 THE BLENDING TECHNIQUE

The Blending technique involves directly mixing nanoscale entities like metal oxide or metal nanoparticles with polymers to create Polymeric Nanocomposite Membranes (PNCMs). Such mixing can be accomplished in two ways, solution blending technique and melt blending technique. Blending techniques are crucial for achieving a homogeneous dispersion of nanoparticles within the polymer matrix.

Solution Blending Technique (SBT)**:** In SBT, polymer and nanoscale structures are dispersed in a common solvent. The process starts by dispersing surface-modified nanoscale entities in the solvent using ultrasonic waves. The resulting mixture is combined with the polymer solution to achieve a uniform dispersion. After solvent evaporation, the intact polymer chains form the nanocomposite matrix. The choice of solvent is crucial, especially for water-soluble polymers, where SBT is more effective. However, the use of toxic and expensive organic solvents poses challenges. . If NCM is to be obtained from polymers insoluble in low-boiling-point solvents, SBT is not preferred.

Melt Blending Technique (MBT): In MBT, nanocomposites are created from the polymer melt. Nanoscale structures and polymers are introduced into an extruder, where they are melted and intensively mixed at high temperatures. Thermal energy is used to enhance polymer chain mobility. MBT is often preferred due to its environmental friendliness and efficiency. However, practical limitations like high temperatures and setup complexity can arise at larger scales. he configuration and design of the extruder and screw influence the quality of nanoscale entity dispersion during blending.

1.5.4 ELECTROSPINNING TECHNIQUE

In the Electrospinning process, a high voltage is applied between a polymer solution droplet and a grounded collector. This creates an electrostatic potential that overcomes the droplet's surface tension, forming a charged liquid jet containing nanoscale entities and dissolved polymer. The jet undergoes thinning, during which solvent evaporates, resulting in the formation of nanofibers that are collected on the collector.

It is a straightforward and efficient method used for producing fibrous membranes. While relatively new, it has gained popularity for various applications such as filtration and desalination, particularly in creating porous membranes. Electrospinning technique is favoured for membrane preparation due to its advantageous features, such as excellent interconnectivity and a relatively even distribution of pores. Its ability to create nanofibers with a large surface area makes functionalization of these membranes easier. The technique offers durability and precise control over membrane structure.

Following gives a step-by-step explanation of the electrospinning process for preparing poly nanocomposite membranes:

- Choose a suitable polymer for electrospinning, ex: polyvinyl alcohol (PVA), polyethylene oxide (PEO), and polyacrylonitrile (PAN). Dissolve the polymer in a suitable solvent to create a polymer solution. The choice of solvent depends on the polymer and its solubility characteristics.
- Incorporate the nanoparticles into the polymer solution. These nanoparticles can be metallic, ceramic, or organic and are chosen based on the desired properties of the nanocomposite membrane.
- Stir the polymer-nanoparticle solution thoroughly to ensure uniform dispersion of nanoparticles within the polymer matrix. This step is crucial to achieve a homogeneous nanocomposite structure.
- Load the polymer-nanoparticle solution into a syringe or a spinneret connected to a high-voltage power supply. Position a grounded or oppositely charged collector (usually a rotating drum or a stationary plate) at a specific distance from the spinneret.
- Apply a high voltage to the polymer solution using the power supply. The electrostatic force generated overcomes the surface tension of the polymer solution. As the polymer solution is ejected from the spinneret, a fine jet is formed and elongated due to the electrostatic repulsion, resulting in the formation of nanofibers.
- The nanofibers are collected on the grounded or oppositely charged collector, forming a nonwoven nanofibrous membrane.

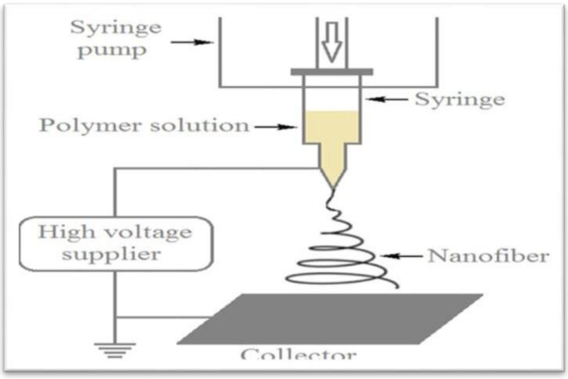


Fig 02: Schematic diagram of the Electrospinning process

1.5.5 TRACK-ETCH PROCESS

The track-etch method is a technique used to manufacture microporous and nanoporous membranes. This method is notable for its ability to produce membranes with precise and uniform pore sizes. A brief overview of the process is discussed below:

- **Irradiation**: A polymer film (often made from materials like polycarbonate or polyethylene terephthalate) is exposed to a high-energy radiation source, such as heavy ions (e.g., uranium, argon, krypton). These ions penetrate the polymer film, creating latent tracks along their paths.

- **Chemical Etching**: The irradiated polymer film is then immersed in a chemical etching solution, typically an acidic or basic solution. The etching process preferentially dissolves the material along the latent tracks, resulting in cylindrical pores where the tracks were formed.

- **Control of Pore Size and Density**: The pore size can be controlled by adjusting the etching time and the concentration of the etching solution. The pore density (number of pores per unit area) is determined by the fluence (number of ions per unit area) during the irradiation step.

1.5.6 MELT PRESSING METHOD

The melt pressing method, also known as melt extrusion or hot pressing, is a technique used to manufacture polymer membranes, particularly microporous membranes, which involves melting a polymer and then forming it into a thin film or sheet, followed by a controlled cooling and stretching process to create pores. A brief description of the melt pressing method is discussed below:

- **Selection of Material:** Commonly used polymers include polyethylene (PE), polypropylene (PP), polyvinylidene fluoride (PVDF), and other thermoplastics. On the other hand, additives like plasticizers, fillers, or porogens (pore-forming agents) are mixed with the polymer to enhance its properties and facilitate pore formation.

- **Melting and Mixing:** The polymer is heated above its melting temperature in an extruder or a heated press. The polymer melt is then thoroughly mixed to ensure uniformity. If additives are used, they are incorporated during this step.

- **Membrane Extrusion or Pressing:** The molten polymer is then extruded through a flat die to form a thin film, or it is placed between heated plates and pressed into a film. The thickness of the film is controlled by adjusting the gap of the die or the pressure applied during pressing.

- **Cooling and Solidification**: The extruded or pressed film is then rapidly cooled to solidify it. Cooling can be done using air, water, or a chilled roll. The cooling rate affects the crystallinity of the polymer, which in turn influences the membrane's mechanical properties and pore structure.

1.6 DIFFERENT CHARACTERIZATION TECHNIQUES FOR POLYMER NANOCOMPOSITE MEMBRANES

Membrane characterization is the process of evaluating and analysing the properties and performance of a membrane. This process involves a range of techniques to understand the membrane's structure, composition, and functional capabilities. Membrane characterization is essential for developing, optimizing, and applying membranes in various industries, including water treatment, gas separation, biotechnology, and pharmaceuticals. There are 2 different types of membrane characterization methods:

1.6.1 STRUCTURAL CHARACTERIZATION TECHNIQUES

This type of characterization focuses on the physical and chemical attributes of the membrane, including its morphology, composition, and surface properties. Techniques used in structural characterization include:

- **Field Emission Scanning Electron Microscopy (FESEM)**: Provides detailed images of the membrane's surface morphology and cross-sectional structure, thereby helping to study the pore distribution and the thickness of the membrane cross section.

- **Contact Angle Measurement**: Determines the wettability of the membrane surface by measuring the angle formed between a liquid droplet and the membrane surface, indicating hydrophobicity or hydrophilicity, a key factor to study the anti-fouling nature of the membranes.

- **Porometry and Mercury Intrusion Porosimetry**: Determines pore size distribution, porosity, and pore connectivity.

- **Transmission Electron Microscopy (TEM)**: Offers high-resolution images of the internal structure at the nanoscale.

- **Atomic Force Microscopy (AFM)**: Measures surface roughness and topography at the nanometre scale.

- **Fourier Transform Infrared Spectroscopy (FTIR)**: Identifies chemical bonds and functional groups within the membrane material.

- **X-ray Diffraction (XRD)**: Analyzes crystalline structures and phase composition.

1.6.2 PERFORMANCE CHARACTERIZATION TECHNIQUES

This type of characterization assesses the functional aspects of the membrane, such as its efficiency, selectivity, flux, permeability and durability in practical applications. Techniques used in performance characterization include:

- **Permeability and Flux Measurements**: Evaluate the rate at which fluids (liquids or gases) pass through the membrane under specific pressure conditions.

- **Selectivity or Rejection Tests**: Measure the membrane's ability to separate or reject certain particles, ions, or molecules from a mixture.

- **Mechanical Strength Testing**: Assesses the membrane's mechanical properties, such as tensile strength, flexibility, and burst pressure.

- **Thermal Stability Analysis**: Uses techniques like Thermogravimetric Analysis (TGA) to study the membrane's stability and performance under varying temperature conditions.

- **Chemical Resistance Testing**: Determines the membrane's stability and performance when exposed to different chemical environments (e.g., acids, bases, solvents).

- **Fouling and Cleaning Studies**: Investigates the membrane's tendency to accumulate contaminants and its ability to be cleaned and restored to its original performance.

1.7 OBJECTIVES OF THE STUDY

A. Synthesis and Characterization of Polymer-Nanocomposite membranes
B. Reduce hydrophobicity by incorporating Nanomaterials
C. Activation and calculation of pore size of Polymeric membranes
D. Application of the Polymeric membrane in methylene blue separation

1.8 OVERVIEW OF THE EXPERIMENTAL STUDY

For our project, synthesis of a nano based polymer membrane was undertaken using the phase inversion technique. Polysulphone, a high-performance polymer, was chosen as the base material, while dimethylacetamide served as the solvent. Titanium dioxide ($TiO2$) and Graphene Oxide (GO) were individually incorporated as the nanomaterial to enhance the polymer's properties, such as Hydrophilicity and finer pore sizes. The integration of TiO2 and GO nanoparticles are expected to impart unique characteristics to the polymer matrix. Additionally, certain structural and performance characterizations were carried to find the applicability of the membrane. The overview of all the materials used, methodologies adopted and characterizations done are mentioned in the table 05, which is discussed in detail in Chapter 03.

TABLE 05: Overview of the materials and procedures used for the study

Membrane Synthesized	Polymer Nanocomposite Organic Membrane
Methodology adopted	Phase Inversion Technique
Polymer Used	Polysulfone (PSf)
Solvent Used	Dimethyl Acetamide (DMAC)
Nanomaterials incorporated	➢ Titanium Oxide (TiO_2) ➢ Graphene Oxide (GO)
Structural Characterizations carried out	➢ FESEM ➢ Contact Angle Measurement ➢ Porosity Measurement
Performance Characterizations carried out	➢ Permeability and Flux Measurements ➢ Selectivity or Rejection Test of MB ➢ Mechanical Strength Testing

1.9 SCOPE OF THE STUDY

This experiment aims to synthesize and characterize polymer-nanocomposite membranes using the phase inversion technique, with a focus on enhancing hydrophilicity and pore size for effective methylene blue separation. Polysulfone (PSf) is used as the base polymer, dissolved in dimethylacetamide (DMAC), with titanium dioxide (TiO2) and graphene oxide (GO) incorporated as nanomaterials to improve membrane properties. The experiment involves structural characterizations, including FESEM, contact angle measurement, and porosity assessment, as well as performance evaluations like permeability, flux measurements, methylene blue rejection, and mechanical strength testing. The findings are expected to provide insights into the applicability of polymeric membranes in water treatment, addressing challenges related to hydrophobicity and fouling, and contributing to advancements in nanocomposite membrane technology.

CHAPTER 02

LITERATURE REVIEW

- ***Muharrem Ince and Olcay Kaplang Ince*** discuss the importance of Nanocomposite Membranes in addressing water scarcity and pollution issues. It highlights the preparation techniques for polymeric nanocomposite membranes - including Interfacial Polymerization (IP), Phase Inversion (PI), Blending Techniques (BT) and Electrospinning Technique (ET). These methods offer various advantages and applications, such as nanofiltration, desalination, etc. It then discusses the Anti-Bacterial and Mechanical properties of Nanocomposite Membranes, to combat biofouling, improve stability, processability, etc.

- ***Yan-Li Ji, Ming-Jie Yin, Quan-Fu An, Cong-Jie Gao*** first, approaches for fabricating polymeric nano-based mixed matrix membranes are summarised, such as surface coating, phase inversion, interfacial polymerization and secondly the manipulation strategies of membrane surface properties, namely, the hydrophilicity/hydrophobicity, surface roughness.

- ***Sivasubramani Divya and Tae Hwan Oh*** developed various polymer nanocomposites membranes for wastewater treatment. They used various nanomaterials like Alumina, GO, Silica, Titanium Oxide and polymers like Polycarbonate, PES, Polypropylene Carbonate, Polylactic Acid, Polyamide to prepare Polymer nanocomposites using different fabrication techniques like Phase Separation, Non-Solvent Induced Phase Separation, Interfacial Polymerization etc. for the purpose of wastewater treatment like removal of industrial dyes, oil, humic acid etc.

- ***Sadaf Noami, Shirin Niroomand , Masoud Rastgar and Mohtada Sadrzadeh*** discuss the application of carbon-based polymer nanocomposite membranes for oily wastewater treatment.

- **Graphene oxide:** *Huang et al.* fabricated ultrafiltration membrane with antifouling property and high stability by coating GO nanosheets on a rough, thin layer of nano porous polyamide.
- **Carbon Nanotubes:** *Saadati* and *Pakizeh* developed novel Polysulphone MWCNTs nanocomposite membrane for Nanofiltration of oil/water emulsions.
- **Carbon Nanofibres:** *Liu et al*. developed macroporous carbon nanofiber films with good flexibility by using purified terephthalic acid to generate micropores inside Electrospun CNF films thereby increasing flexibility by reducing stress.

- ***Jonathan M. Schuster, Carlos E. Schvezov, Mario R. Rosenberger*** discuss the application of the sessile drop method, a quick and straightforward technique for determining the contact angle between solids and liquids. This angle is crucial for understanding surface characteristics, specifically the wettability of solid surfaces by liquids, and for calculating surface free energy. The study investigates the impact of drop volume, elapsed time between drop placement and measurement, and substrate cleaning methods (no cleaning, ethanol cleaning, acetone cleaning) on the contact angle of Ti6Al4V and AISI 316 stainless steel.

- ***Jaewoo Lee, Hee Ro Chae*** developed a Graphene Oxide nanoplatelets composite membrane for wastewater treatment by using Polysulphone, Graphene oxide from Graphite by phase inversion process. They found the membrane with high water flux, antifouling property.

- ***Muhammad Ayaz, Amir Muhammad , Muhammad Yuanas, Asim Laeeq Khan, Mashallah Rezakazemi*** synthesized polysulfone membranes integrated with Alumina Nanoparticles using phase inversion to enhance water treatment efficiency. Various concentrations of alumina were tested but the membrane with 0.1% concentration of alumina demonstrated superior performance.

- ***Hadi Hosseini, Abbas Zirakjou, David Julian McClements, Vahabodin Goodarzi, Wei-Hsin Chen*** synthesized environmentally friendly nanocomposite hydrogels which were reinforced with different concentrations of graphene oxide nanoparticles. It was found that with increase in concentration of nanoparticles there was a decrease in the pore size of the membranes.

- ***Yang Zeng , Xiangtao Tang , Yan Qin , Adila Maimaiti , Xudong Zhou , Yujie Guo , Xin Liu , Wenqing Zhang , Jie Gao , Lingfan Zhang*** developed an carboxymethyl/ alginate cellulose-melamine sponge composite with high porosity and enhanced adsorption performance for methylene blue dye.

- ***Sayed Mohsen Hosseini, Ehsan Ba gheripour, Mohsen Ansari*** synthesized a new mixed matrix polyethersulfone -co-Magnesium oxide nano composite nanofiltration membrane by phase inversion method using DMAC as solvent.It was observed that the porosity of the membranes reduces on increasing the concentration of nanomaterials. Moreover the surface hydrophilicity was seen to increase proportionally with the increase in nanomaterial concentration.

CHAPTER 03

MATERIALS AND FABRICATION TECHNIQUES

3.1 MATERIALS USED

The choice of polymer-solvent combinations for making poly-nanocomposite membranes depends on the specific polymer and nanoparticles selected, as well as the desired properties of the final membrane. Different polymers have different solubility characteristics, and the choice of solvent is crucial for achieving good dispersion of nanoparticles and uniform membrane formation. The choice of solvent should also consider environmental and safety considerations. Additionally, it may be needed to optimize the polymer concentration, nanoparticle loading, and other process parameters to achieve the desired membrane properties.

TABLE 06: Commonly used polymer-solvent-nano combinations for making polynanocomposite membranes are given below:

Polymer	Solvents	Nanomaterials
Polysulphone	N-methyl-pyrrolidone(NMP), dimethylformamide(DMF), dimethylacetamide (DMAc)	graphene oxide, carbon nanotubes, metal oxides
Polyvinylidene fluoride	N-methyl-pyrrolidone(NMP), dimethylformamide(DMF), dimethylacetamide (DMAc), acetone	Titanium Oxides Zinc Oxide alumina nanoparticles.
Polyacrylonitrile (PAN)	Dimethyl sulfoxide(DMSO) dimethylformamide (DMF)	Carbon nanotubes Graphene Metal oxide nanoparticles
Polyether sulfone (PES)	N-methyl-pyrrolidone(NMP), dimethylformamide(DMF), dimethylacetamide(DMAc) tetrahydrofuran (THF).	Carbon nanotubes silica nanoparticles clay nanoparticles.

3.1.1 POLYMER: Polysulphone

The membranes prepared for this research primarily focuses on Wastewater treatment, which makes Polysulphone polymer one of the most suitable choices as the Base Polymer. Polysulphone based membranes have been widely used for wastewater treatment due to their desired properties like stability, high mechanical strength and ease of modification. Polysulphone is a high-performance engineering thermoplastic known for its excellent thermal stability, chemical resistance, and mechanical properties. These characteristics make it a suitable candidate for membrane applications.

The choice of Polysulphone offers several advantages in the context of membrane synthesis, especially when creating nanocomposite membranes:

- **High Temperature Resistance:** Polysulphone exhibits good thermal stability and can withstand relatively high temperatures. This is advantageous for applications where membranes may be subjected to elevated temperatures, such as in processes involving hot gases or liquids.
- **Chemical Resistance:** Polysulphone is known for its excellent chemical resistance. It can withstand exposure to a wide range of chemicals without significant degradation. This property is crucial in applications where the membrane comes into contact with various substances, such as in water treatment or chemical processing.
- **Mechanical Strength:** Polysulphone membranes generally possess good mechanical strength and durability. This is essential for maintaining the structural integrity of membranes, especially in processes involving pressure differentials or mechanical stresses.
- **Membrane Pore Structure:** Polysulphone membranes can be easily fabricated to have a well-defined and controlled pore structure. This is important for achieving the desired separation properties, whether it involves microfiltration, ultrafiltration, or other membrane-based processes.
- **Compatibility with Nanoparticles:** Polysulphone is compatible with a variety of nanoparticles commonly used in nanocomposite membrane fabrication, such as metal oxides or carbon-based materials. This compatibility allows for the effective incorporation of nanoparticles into the polymer matrix, leading to enhanced properties of the resulting nanocomposite membrane.

- **Biocompatibility:** Polysulphone is known for its biocompatibility, making it suitable for applications in biotechnology and medical fields. When used in the fabrication of nanocomposite membranes, this property can be beneficial for applications such as protein separation or medical filtration.
- **Hydrophilicity and Hydrophobicity Balance:** Polysulphone membranes often exhibit a good balance between hydrophilic and hydrophobic characteristics. This can be advantageous in applications where controlled permeation of water or other fluids is required, as the membrane can be tailored to be selective to specific molecules or ions.

Fig 03: Polysulphone Polymer

3.1.2 SOLVENT: Dimethyl-acetamide (DMAC)

Dimethylacetamide (DMAc) is a polar aprotic solvent that is commonly used in the preparation of polymeric membranes, including poly nanocomposite membranes. Its specific properties make it suitable for certain polymer systems and processes.

Using Dimethylacetamide (DMAc) as a solvent in the preparation of poly nanocomposite membranes with Polysulphone offers several advantages:

- **High Solvating Power:** DMAc has a high solvating power, which means it can dissolve a wide range of polymers, including Polysulphone. This facilitates the preparation of homogeneous polymer solutions, ensuring uniform distribution of the polymer in the membrane.

- **Good Polymer Solubility:** Polysulphone exhibits good solubility in DMAc, allowing for the easy preparation of concentrated polymer solutions. This is crucial for achieving a high polymer content in the casting solution, leading to the formation of membranes with desirable properties.
- **Controlled Film Formation:** DMAc allows for controlled film formation during the casting process. This solvent provides the necessary viscosity and wetting properties for the casting solution, allowing for the creation of a thin and uniform polymer film on the substrate.
- **Enhanced Pore Formation and Morphology Control:** The use of DMAc in the casting solution can lead to enhanced pore formation and morphology control in the resulting membranes. This is important for tailoring the membrane structure to achieve specific separation properties.
- **Facilitates Nanoparticle Dispersion:** DMAc is often effective in dispersing nanoparticles in the polymer solution. This is important when incorporating nanoparticles into the polymer matrix to form nanocomposite membranes. The solvent's ability to disperse nanoparticles uniformly can enhance the overall properties of the membrane.
- **Low Toxicity:** DMAc is considered to have relatively low toxicity compared to some other solvents used in membrane fabrication. This is an important consideration for safety and environmental concerns during the manufacturing process.
- **High Boiling Point:** DMAc has a relatively high boiling point, which aids in the slow evaporation of the solvent during the membrane formation process. This slow evaporation helps prevent the formation of defects in the membrane and allows for better control over the membrane structure.

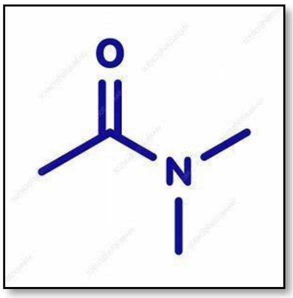


Fig 04: Structure of Dimethylacetamide

3.1.3 NANOMATERIALS: Titanium dioxide and Graphene oxide

Nanomaterials refer to materials with dimensions on the nanoscale, typically ranging from 1 to 100 nanometres. These materials exhibit unique and often enhanced properties due to their small size and high surface-to-volume ratio. In the context of polymeric membranes, nanomaterials are incorporated to create poly nanocomposite membranes, where nanoparticles are dispersed within the polymer matrix. This incorporation is driven by the desire to improve the performance and functionality of the membranes. Nanomaterials can impart several advantageous features, such as enhanced mechanical strength, improved thermal stability, and increased resistance to chemical degradation. Additionally, nanoparticles have been added in our research majorly with the purpose to modify the membrane's surface properties, leading to changes in hydrophilicity, hydrophobicity, and surface charge.

When preparing poly nanocomposite membranes using the Polysulphone polymer in combination with the Dimethylacetamide (DMAc) solvent, various nanomaterials can be incorporated to enhance the membrane's properties, such as Graphene and Graphene Oxide nanomaterials, Titanium or other metal oxides, Carbon Nano-tubes (CNTs), etc. The choice of nanomaterial depends on the targeted application and the desired properties of the resulting membrane.

For this project, we have tried two combinations: One using Titanium dioxide Nanomaterials and the other using Graphene Oxide Nanomaterials, incorporated into the Polysulphone-Dimethylacetamide Polymer-Solvent Solution.

A. The incorporation of titanium dioxide (TiO_2) nanomaterials into polymeric membranes, especially when using a Polysulphone and dimethylacetamide (DMAC) solvent combination, offers the following major benefits:

- **Improved Hydrophilicity:** TiO_2 nanoparticles can enhance the hydrophilic nature of the polymeric membrane. This modification can lead to improved water permeability and flux, making the membrane more efficient in water treatment and filtration processes.
- **Selective Separation Properties:** The presence of TiO_2 nanoparticles can influence the selectivity of the poly nanocomposite membrane. Depending on the nanoparticle size and distribution, the membrane can exhibit specific

separation properties, making it suitable for applications like gas separation or molecular filtration.

- **Reduced Fouling and Scaling:** TiO_2 nanoparticles can help reduce fouling and scaling on the membrane surface. The photocatalytic activity of TiO_2 may assist in breaking down foulants and preventing their accumulation, leading to longer membrane lifespan and improved performance.
- **Improved Mechanical Strength:** Titanium oxide nanoparticles can enhance the mechanical strength and stability of the polymeric membrane. The incorporation of TiO_2 nanomaterials helps to reinforce the polymer matrix, making the resulting nanocomposite membrane more robust and resistant to deformation.

B. Incorporating graphene oxide (GO) nanomaterials into Polysulphone membranes prepared using Dimethylacetamide (DMAc) as a solvent can offer several advantages, resulting in poly nanocomposite membranes with enhanced properties. Here are some key advantages of using GO in combination with PSU and DMAc:

Improved Hydrophilicity: Graphene oxide has hydrophilic functional groups on its surface, which can enhance the hydrophilicity of the membrane. This is beneficial for applications like water filtration, where a higher affinity for water molecules is desired.

Antibacterial Properties: Graphene oxide has been reported to exhibit antibacterial properties. Incorporating GO into poly nanocomposite membranes can potentially impart antibacterial characteristics, making the membrane suitable for applications where microbial growth needs to be controlled.

Mechanical Strength and Stability: Graphene oxide is known for its exceptional mechanical properties, including high tensile strength and modulus. Incorporating GO into the Polysulphone matrix can enhance the mechanical strength and stability of the resulting poly nanocomposite membrane, making it more robust and durable.

Improved Thermal Stability: Graphene oxide has high thermal stability. When incorporated into Polysulphone membranes, it can contribute to improved thermal resistance. This is especially beneficial in applications where the membrane is exposed to elevated temperatures, as it can help maintain the structural integrity of the membrane.

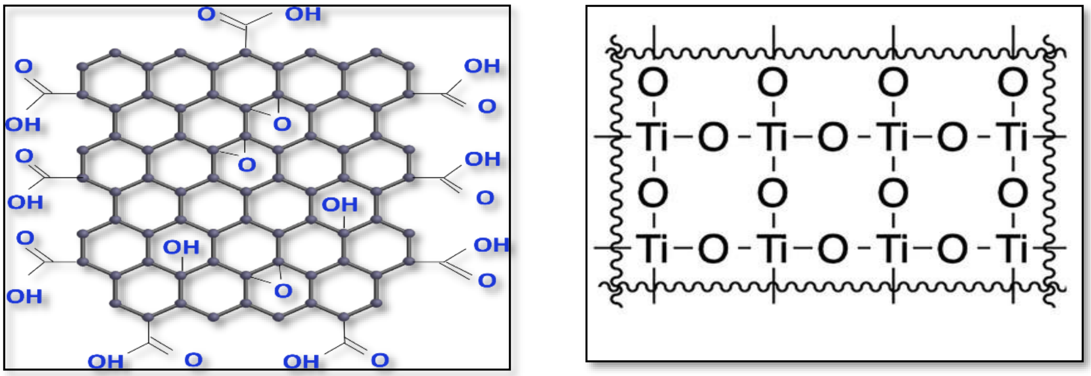


Fig 05: Structure of Graphene oxide and titanium dioxide

3.2 FABRICATION TECHNIQUE USED

Rapid global population growth and industrial activities have led to water scarcity, particularly in arid regions. The scarcity affects over a billion people, with water pollution from various sources compounding the issue. Notably, toxic heavy metals pose severe threats to ecosystems and human health, motivating the need for effective water treatment. Membrane technologies, like nanocomposite membranes (NCMs), have emerged as crucial tools for pollutant removal. By incorporating nano-sized materials, such as carbon nanotubes and metal oxides, into polymer matrices, NCMs offer advantages like enhanced permeability, selectivity, and fouling resistance. Polymeric nanocomposite membranes (PNCMs) further amplify these benefits, with their potential highlighted by a growing body of research. This technology's energy efficiency, eco-friendliness, and adaptability make it a promising avenue for addressing water scarcity and contamination challenges.

PHASE INVERSION PROCESS

The phase inversion process, used for the membrane’s synthesis is a common method for manufacturing polymer membranes, including poly-nanocomposite membranes. It involves the transformation of a homogeneous polymer solution into a solid membrane structure through a change in the thermodynamic conditions, typically induced by solvent removal.

This transformation process can be performed using three techniques: Immersion Precipitation, where the polymer solution is immersed in a coagulation bath to allow the exchange of solvents to occur, which has been used for this research project. The other two techniques are thermally induced phase separation, in which the de-mixing process occurs by subjecting the membrane to high temperatures, and evaporation induced phase separation, which occurs through the evaporation of the volatile solvent used to prepare the polymer solution of interest.

Here's an overview of the phase inversion process used for the manufacturing of polynanocomposite membranes:

- Begin by preparing a polymer solution by dissolving the polymer in a suitable solvent. The choice of solvent depends on the polymer and its compatibility with the nanofillers. For this project, polymer used is Polysulphone in Dimethylacetamide (DMAC) solvent.
- Disperse the nanofillers (such as nanoparticles, nanotubes, or nanosheets) in the polymer solution. Titanium Oxide and Graphene Oxide nanoparticles have been used for study in this project. Achieving a uniform dispersion is crucial for enhancing the properties of the final nanocomposite membrane.
- Ensure thorough mixing to achieve a homogeneous blend of the polymer and nanofillers in the solution. Various mixing techniques, such as magnetic stirring or ultrasonication, may be employed. For ease of processing and minimizing cost, magnetic stirrer has been used for this particular project.
- The polymer-nanofiller solution is cast or applied onto a substrate using methods like doctor-blading, spin-coating, or dip-coating
- Allow the solvent to evaporate from the cast solution. This can be achieved through air drying, controlled heating, or a combination of both. As the solvent evaporates, the polymer undergoes phase separation.
- During solvent evaporation, the polymer-solvent-nanofiller system undergoes phase separation, leading to the formation of two phases: a polymer-rich phase and a solvent-rich phase. This phase separation is a critical step in creating the desired membrane structure. The phase-separated structure evolves into the final membrane structure as the solvent continues to evaporate.
- After allowing the solvent to evaporate, the cast membrane is then immersed in a quenching or coagulation bath to enhance phase separation and solidification.

- Rinse the formed membrane to remove any residual solvent or by-products, followed by immersing the formed membrane in a deionized water bath for 1224 hours. Subsequently, dry the membrane to obtain the final polynanocomposite membrane.

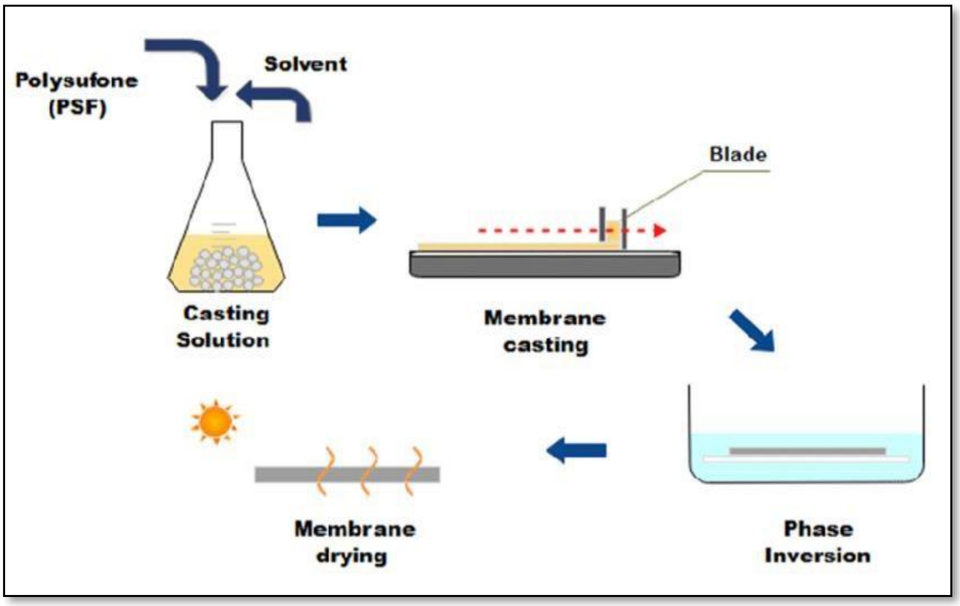


Fig06: Schematic Diagram of Phase Inversion process

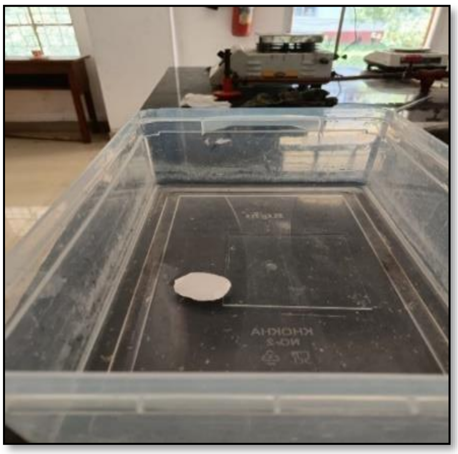


Fig 07: Membrane quenching bath

Fig 08: Magnetic stirrer setup

3.3 MEMBRANE COMPOSITION TRIAL AND ERROR

Initially, Acetone was chosen as the solvent for Polysulphone, but it was discovered that they were not compatible due to the low solubility of Polysulphone in acetone. Following several trials with alternative solvents, such as dimethylformamide (DMF), it was determined that Dimethylacetamide was the optimal solvent for Polysulphone, forming a compatible solvent-polymer combination.

The subsequent step involved optimizing the concentration of the Polysulphone polymer. Initially set at 10%, the concentration was incrementally increased to 12%, 14%, and finally 16%. It was observed that membranes with polymer concentrations of 12% and 14% exhibited superior uniformity, robustness, and physical strength.

Moving forward, the focus shifted to determining the concentration of nanomaterials. For Graphene Oxide-based polymeric membranes, an initial composition of 2% Graphene Oxide revealed solubility challenges. Subsequently, membranes were prepared with concentrations of 1%, 0.5%, 0.3%, and 0.25% for both 12% and 14% polymer concentrations. The findings indicated that membranes with 14% polymer concentration, incorporating either 0.3% or 0.5% Graphene Oxide, demonstrated optimal membrane properties.

In parallel, Titanium Oxide was employed as a nanofiller and tested similarly to Graphene Oxide. The results suggested that a polymer concentration of 14%, infused with 0.3% or 0.25% Titanium Oxide, yielded the most favourable membranes.

TABLE 07 : Membranes synthesized of different compositions

Sample	Polymer(%) (Polysulfone)	Solvent(%) (Dimethylacetamide)	Nanomaterial(%)
01	14	86%	0
02	14	85.7%	Graphene oxide-0.3%
03	14	85.7%	Titanium dioxide-0.3%

3.4 BATCH CELL SETUP

A batch cell is typically an instrument where a membrane is placed inside it and water is made to flow through the cell. This helps us to find the porosity of the membrane using the Hagen–Poiseuille equation. Water would be collected in a certain amount which will help to find the flux generated. An external pressure will be applied under which the water will flow through the membrane.

3.4.1 MODIFICATIONS MADE IN THE BATCH CELL DESIGN

1st System Setup

The first trial for batch cell setup consisted of two ply boards with a hole in the middle. Pipes made from PVC was fixed on one side of the board. The hole made in the ply board had the same diameter as the PVC pipe. After the pipes had been fixed, flex and M-seal was used so that any kind of leakage can be prevented. After that both sides of the ply boards were joined together and holes were made around the ply boards so that both boards can be screwed together. The picture of the setup is given below:

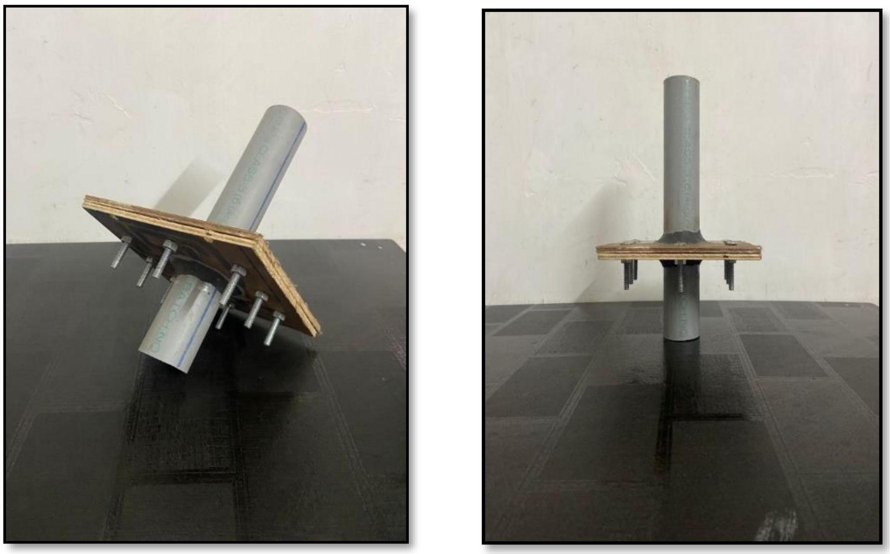


Fig 09: Batch cell first trial

Problems Faced

After flowing water through the top of the cell it was found that water was leaking from the small gap that remained while screwing the ply board. Also, water was leaking from the screw holes that were made. Main issue was that the ply board surfaces weren't smooth enough. Water flow through those irregularities. Even after trying to make the surface as smooth as possible by mechanical means water leakage couldn't be solved. So, after multiple trials this setup was discontinued.

2nd System Setup

The second trial was made with a torch light. A typical steel torch has a hollow structure from which water can be made to flow if all the holes are sealed off. A torch light was modified into a batch cell where the bottom portion of the torch would be used to place the membrane for water to flow through it. Pressure can be given using a nitrogen cylinder through the top portion of the torch. A small hole was made so that the pipe through which pressure would be given can be inserted.

The images of the torch setup is given below:

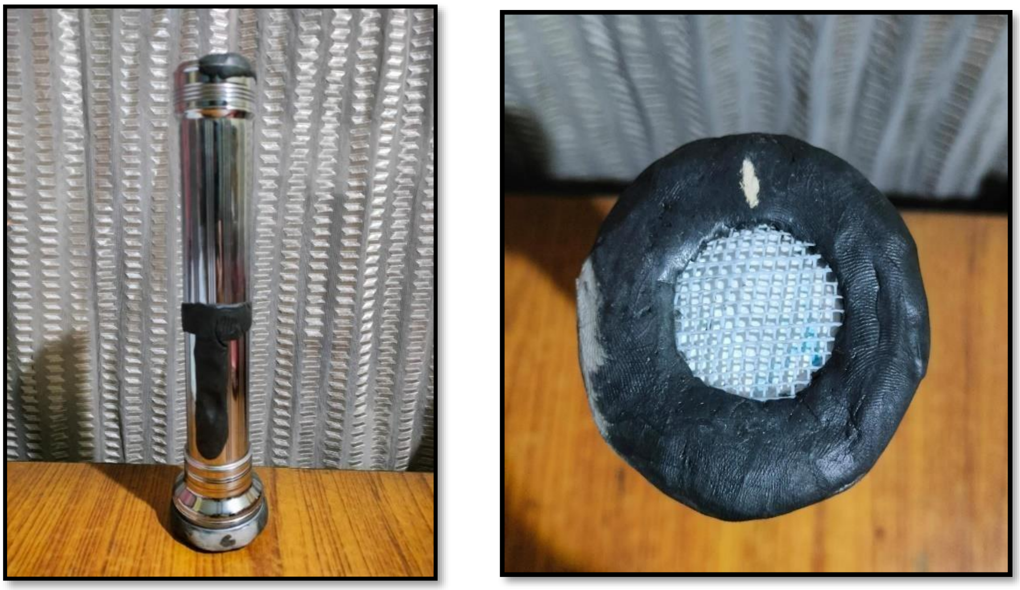


Fig 10: Batch cell second trial

Challenges Faced

The main issue here was also about leakage of water. Water flows through the bottom section of the torch where the membrane is placed. Various trials were made to stop the leakage but no fruitful results could be found.

Another interesting problem that we encountered is that the cross-section of the membrane was too large to withstand the external pressure that was applied. Merely a pressure of about 0.2 psi led to the rupturing of the membrane. This happened mainly because the thickness of the membrane that was synthesizes was very less, almost like a paper. Thus, applying a high pressure on a large area ruptures the membrane. Hence, for checking the porosity a smaller cross-sectional area would be required.

3rd System Setup

The third trial was using standard iron piping components. It consists of one square plug fig where membrane will be placed along with two double sided threaded nipple. One nipple is if 9inch and other one is of about 2inch. Water will be flown through the longer nipple flowing through the membrane that was placed in the square plug fig and pass out through the smaller nipple. The top portion of the longer nipple is welded and only a small hole is made at the top for applying external pressure. The image of this batch cell is given below:

Fig 11: Batch cell third trial

Challenges Faced

Issue of leakage was not yet solved with this setup. Also, there was no space for packing material which would hinder the uniform distribution of pressure throughout the system.

4th System Setup

The last and final trial was done with the same setup as above but instead of iron, PVC material was being used. The top portion of the pipe was covered with a pipe end cap to completely seal the pipe. A small hole has been made on the top to insert a thin pipe which would provide the required pressure. The cap of the setup is filled with packing material. It is caged using a fabricated polymer net tightly so that it doesn't fall off when pressure is applied. Photo of complete setup is show. This setup ensures 100% leak resistance along with a well-supported packing material for uniform pressure distribution.

Fig 12: Final Batch cell setup

3.5 MEMBRANE CHARACTERIZARIONS CONDUCTED

3.5.1 FESEM FOR MEMBRANE CHARACTERIZATION

OBJECTIVE:

- To observe morphology and changes of membrane surface before and after nanoparticle incorporation into the membrane matrix,
- To identify whether the synthesized membranes are porous
- To check the thickness of membranes and the effect of nanoparticles on the overall porosity of the prepared membranes (Section 3.5.2)

Field Emission Scanning Electron Microscopy is an advanced microscopy technique that uses a focused beam of electrons to achieve high-resolution images of the surface of a sample. The following are the key components and principles of FESEM:

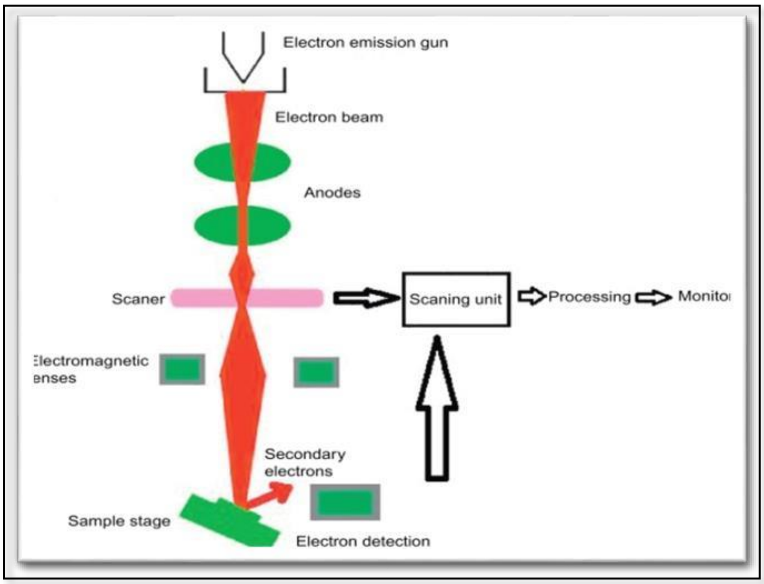


Fig 13 : Schematic representation of FESEM

Electron source:

- FESEM uses a field emission electron source, where electrons are emitted from a sharp metallic tip under the influence of a high electric field.
- The use of field emission allows for a smaller electron source and higher spatial resolution compared to traditional scanning electron microscopy (SEM).

Electron Optics:

- The emitted electrons are accelerated and focused using a series of electromagnetic lenses and apertures.
- Magnetic lenses play a crucial role in focusing the electron beam to achieve high resolution.

Sample Preparation:

- Samples for FESEM must be conductive or coated with a thin layer of conductive material (e.g., gold or carbon) to prevent charging effects. Sample preparation is a critical step, and the surface must be clean and dry to ensure accurate imaging.

Scanning Process:

- The electron beam scans the surface of the sample in a raster pattern.

Image Formation:

- The signals generated by the interaction of electrons with the sample are collected and used to form an image.
- Secondary electrons provide topographical information and are commonly used for high-resolution imaging of surface features.
- Backscattered electrons provide compositional contrast, as they are sensitive to the atomic number of the elements in the sample.

Detectors:

- Different detectors can be used to capture specific signals.
- FESEM offers high spatial resolution, typically in the range of nanometres or even sub-nanometres. This is due to the small electron source and advanced electron optics.

The membranes were characterized using the Cross Sectional Morphology to determine its Thickness. Image J software was then used to determine the accurate thickness of the membrane at various angles in the Cross Section.

3.5.2 MEMBRANE POROSITY

The porosity of membranes refers to the extent of empty spaces or voids within the structure of a membrane material. Membranes are thin, selective barriers that separate different phases or components within a system, such as liquids or gases. Porosity in membranes is a critical factor as it influences their permeability and selectivity, which are essential properties for various applications.

Membrane porosity refers to the void volume fraction of the membrane. It is defined as the ratio of pore volume to the total volume of the membrane. The surface porosity of the membrane is calculated on the basis of dry wet using :

$$Overall\ porosity\ (\%) = \frac{w_w - w_d}{A\ X\ \delta \times\ \rho_\omega}$$

Where, W_w and W_d are weights of wet and dry membranes (in grams) respectively.

A is the surface area of the membrane sample (in cm^2)

δ is the thickness of the membrane sample (in cm)

ρ_w is the density of pure water (gm/cm^3)

3.5.3 CONTACT ANGLE EXPERIMENT

OBJECTIVE:

- To check the hydrophilic / hydrophobic nature of the polymeric membranes.

Wettability, the extent to which a liquid interacts with a solid surface, is a critical characteristic in materials science, and its measurement is often determined by the contact angle (CA). This angle is a quantitative indicator of how a liquid, particularly water in this context, behaves on a given surface. The distinction between hydrophilic and hydrophobic surfaces, where water spreads easily or beads up, respectively, can be made based on the contact angle measurement. Moreover, the contact angle is not merely a descriptive metric; it serves a practical purpose.

THE SESSILE DROP METHOD

The primary technique employed for measuring the contact angle (CA) is the sessile drop method. This method involves placing a drop of liquid on the surface of a material, creating a system with three distinct phases: solid (representing the solid surface), liquid (the droplet), and gas (the surrounding atmosphere). Within this system, three interfaces emerge - liquid-vapor (drop-atmosphere), solid-liquid (solid material-drop), and solid-vapor (solid material-atmosphere) interfaces. The point where these interfaces intersect forms a line known as the three-phase line or contact line, as depicted in Figure. *The contact angle is precisely defined as the angle formed by the tangent of the liquid-vapor interface and the solid-liquid interface at this contact line.*

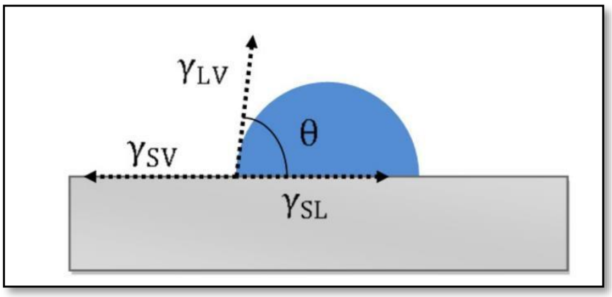


Fig 14 : Schematic of a contact angle of a liquid drop on a solid surface

To quantify the contact angle, physicist Thomas Young proposed a simple equation that relates it to the interfacial tensions within the system:

$$\boxed{\cos\theta = \frac{\gamma^{sv} - \gamma^{sl}}{\gamma^{lv}}}$$

Here, θ is the contact angle, γsv is the solid-vapor surface tension, γsl is the solid-liquid surface tension, and γlv is the liquid-vapor surface tension. This equation reflects the balance of forces at the three-phase line and offers a fundamental relationship between the contact angle and the interfacial tensions of the system. The sessile drop method, along with such mathematical models, provides a robust approach for understanding and quantifying the wetting behaviour of liquids on solid surfaces in various scientific and industrial applications.

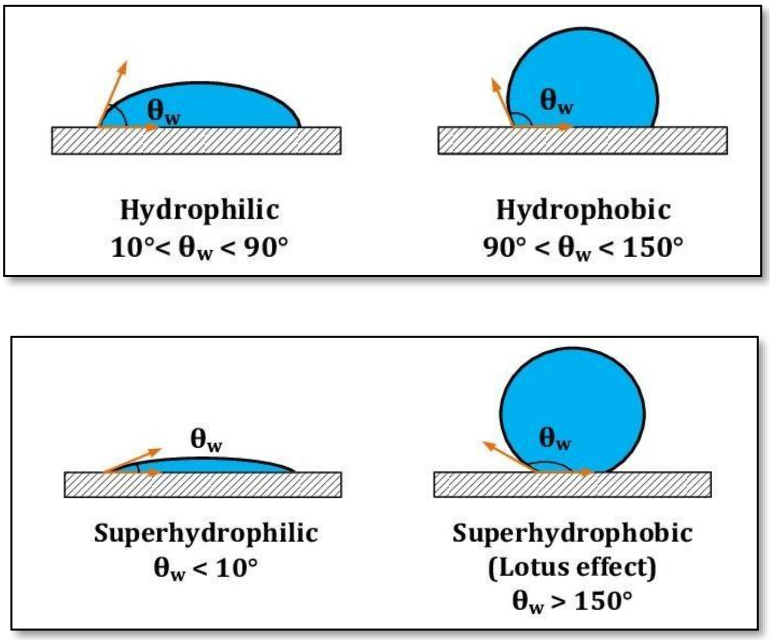


Fig 15: Categories of Wetting

Based on surface properties, there are two types of polymeric membranes: Hydrophobic repels water and allows oil droplets to permeate freely which may lead to fouling issues. Hydrophilic allows water to transfer and repels oil droplets, which exhibits better antifouling issues. Membrane fouling is a process by which the particles, colloidal particles, or solute macromolecules are deposited or adsorbed onto the membrane pores or onto a membrane surface by physical and chemical interactions or mechanical action, which results in smaller or blocked membrane pores. In our project, we aim to produce hydrophilic polymeric membrane by incorporating the nanomaterial so to reduce the fouling issues which may cause severe flux drops and affects the quality of water produced.

Several factors have been reported influencing the experimental determination of the contact angle by the sessile drop method and must be considered:

- **Drop Size:** The literature suggests that the volume of the liquid drop, or drop size, should fall within the range of 0.5 to 10 mm^3 according to various studies. While smaller drops are generally used, it is emphasized that when comparing results obtained with drops of different sizes, the effect of drop size should be considered.

- **Time Since Deposition**: The measurement of the contact angle should be conducted shortly after the deposition of the drop on the solid surface. Maintaining a consistent time interval for all measurements helps minimize the impact of volume loss due to evaporation and potential reactions between the liquid and the surface.

- **Temperature:** Small changes in temperature within the range of 20 to 40 ºC are noted to have little influence on the surface tension of the liquid. Consequently, such minor temperature variations are considered to have no appreciable impact on the contact angle measurement.

- **Mechanical Vibration:** To prevent deformation of the liquid, drop due to vibrations, it is emphasized that the sample should remain static during the measurement. Mechanical vibrations could introduce variability and inaccuracies into the results, underscoring the need for a stable experimental setup.

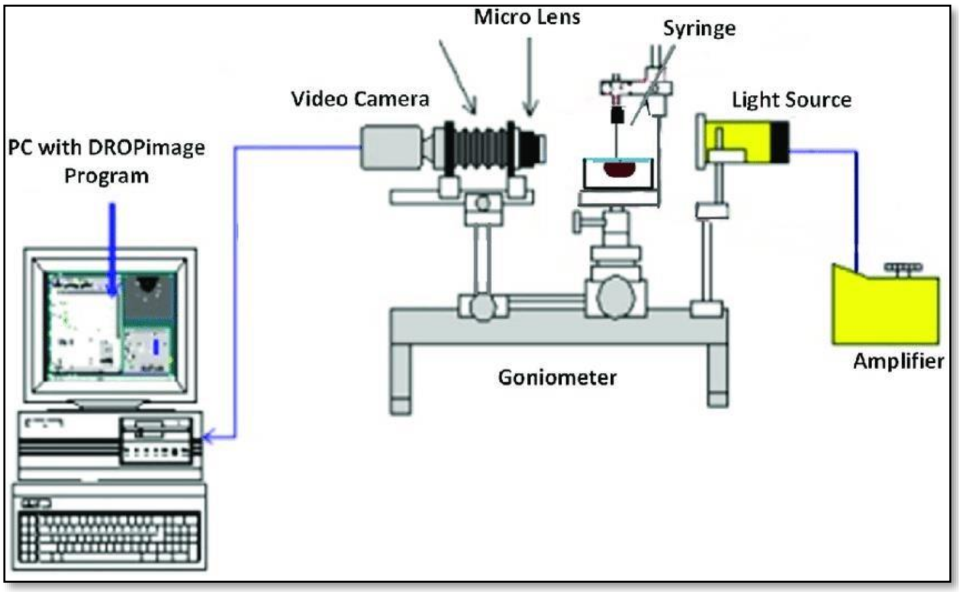


Fig 16 : Goniometer (to measure contact angle)

A contact angle goniometer is an instrument that measures the contact angle of a droplet on a surface. This is a useful, indirect measurement of surface wetting. The sample is cleaned with detergent to remove any contaminants. It is then rinsed with water to eliminate detergent residues. Further rinsing with ethanol is done. The sample is dried using hot air. Once the sample reaches room temperature, a very precise volume of liquid is dosed onto the material. A graduated syringe is used for accurate dosing. The liquid is added slowly and carefully to ensure the formation of a regular and symmetrical drop. After placing the drop on the material, a photograph is taken. The photograph is processed digitally.

The software ImageJ is used for analysing the photograph. Two angles are measured on the photograph: the left and the right angles. The mean of both angles is calculated. A symmetry test is performed to ensure a single contact angle.

3.5.4 CONSTANT PTRSSURE ACTIVATION OF MEMBRANES

The integration of nanoparticles into polymer matrices enhances membrane properties, including mechanical strength, Hydrophilicity, permeability among others. Activation of these membranes under constant pressure is a critical process influencing their performance and efficiency.

It is a process where a fixed pressure is applied to the membrane, ensuring uniform pore size distribution and enhanced membrane properties. Application of constant pressure leads to membrane compaction, reducing pore size variability and enhancing selectivity. Activation leads to the Nanoparticles creating microchannels, improving permeability.

The activation process under constant pressure is carried out in the batch cell setup, containing the membrane, which is fed with 150 ml of water from the top. A pressure of 25 psi (for sample 03) and a pressure of 18 psi (for sample 01) is applied to activate both the respective Membranes. The weight of the permeate is recorded after an interval of 60 seconds each, until the difference between two consecutive weight readings becomes concurrent. The readings for constant pressure activation for both the membranes (sample 01 and sample 03) are discussed in Section 4.3.

3.5.5 PORE SIZE DETERMINATION

Once the Membranes have been activated, we can now calculate the flux through the membrane and then find the average pore size of the membrane using the Darcy's Law and Hagen Poiseuille's Equation. For this experiment, we feed 100 ml of water from the top of the batch cell, whose flux is to be calculated. Inside the batch cell, we fit the membrane and pressurize the water through it, at 3 different pressures, as depicted in the tables below. The weight of the permeate is calculated, from which the flux can be obtained, using the formula:

$$\textbf{VOLUMETRIC FLUX} = \frac{\textbf{MASS X DENSITY}}{\textbf{AREA X TIME}}$$

For our setup, D = 1 inch = 2.54cm = 0.0254 m,

AREA = $\frac{\Pi D^2}{4} = 5.07 \times 10^{-4}$ m^2

3.5.6 CALIBRATION OF THE METHYLENE BLUE CURVE

Methylene blue, a versatile compound used in medicine, biology, and chemistry, is commonly found in industrial effluents. Despite its beneficial applications, it can have harmful effects, High doses may cause methemoglobinemia, while it poses a neurotoxicity risk, leading to serotonin syndrome when combined with certain drugs. It can induce haemolytic anaemia in individuals with G6PD deficiency and cause hypersensitivity reactions, blood pressure changes, and gastrointestinal distress. Additionally, methylene blue may cause skin and urine discoloration, local tissue damage at injection sites, and potential teratogenic effects during pregnancy

For our research, we want to determine the percentage of Methylene Blue Removed from a known solution, in order to estimate the rejection of Methylene Blue achieved from its's solution, for which we prepare a Calibration Curve. Calibration curve is a plot used to determine the concentration of a substance in an unknown sample by comparing it to a set of standard samples of known concentration. The curve is plotted with the concentration of the standards on the x-axis and their corresponding absorbance values on the y-axis.

To obtain the Calibration Curve, we prepare a series of known solutions of methylene Blue. Using a UV-Vis spectrophotometer, we measure the absorbance of each standard solution at the maximum absorption wavelength for methylene blue, at 660 nm. Plot the absorbance values (y-axis) against the concentrations (x-axis) to generate the calibration curve.

3.5.7 METHYLENE BLUE REJECTION EFFICIENCY OF MEMBRANES

An aqueous solution of Methylene Blue having concentration of 100 ppm was used as the feed, which was fed to the Batch Cell, containing the membrane (only sample 3 used for this purpose, as it has superior properties) at a nominal pressure of 10 psi, to study the amount of Methylene Blue that was absorbed from the solution by the membrane. The permeate was then collected in different beakers for a period of 4 minutes each, for 3 independent cycles, to study the reduction in the membrane's efficiency to remove MB from the feed solution. The absorbance of the feed was then identified using UV-Vis Spectrometer, and correspondingly it's concentration from the Calibration Curve. The MB Rejection Percentage was calculated using the formula given as:

$$\boldsymbol{R}\left({}^{0}/_{0}\right) = \frac{\boldsymbol{C_F} - \boldsymbol{C_P}}{\boldsymbol{C_F}}\ \boldsymbol{X}\ \mathbf{100\%}$$

where, R is the Rejection of MB from the Solution in %, C_F is the concentration of Methylene Blue in the feed (100 ppm), and c_p is the Concentration of Methylene Blue in the Permeate.

Fig 17: Methylene Blue Solution of Different Concentrations

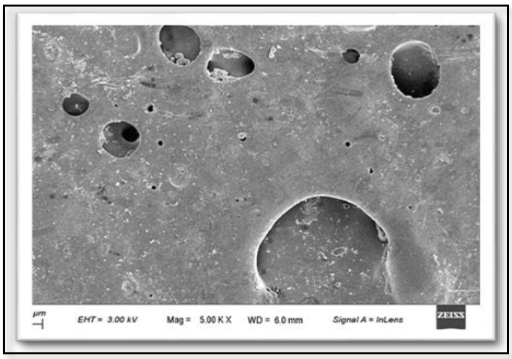
CHAPTER 04

RESULTS AND DISCUSSIONS

4.1 OBSERVATIONS FROM FESEM

4.1.1 Morphology and Porosity of the polymeric membranes

The FESEM experiment for our membrane characterisation was performed at Indian Institute of Technology, Guwahati .The SEM Images obtained (as in the figure below) for various compositions of the Membranes indicates the presence of pores in each membrane, with variations with addition of different composition and type of nanomaterial used .



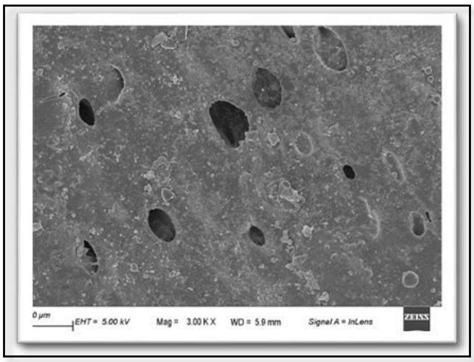


Fig18: FESEM image for Sample 01

(without nanomaterial)

Fig19: FESEM image for Sample 02

(with 0.3% Graphene oxide)

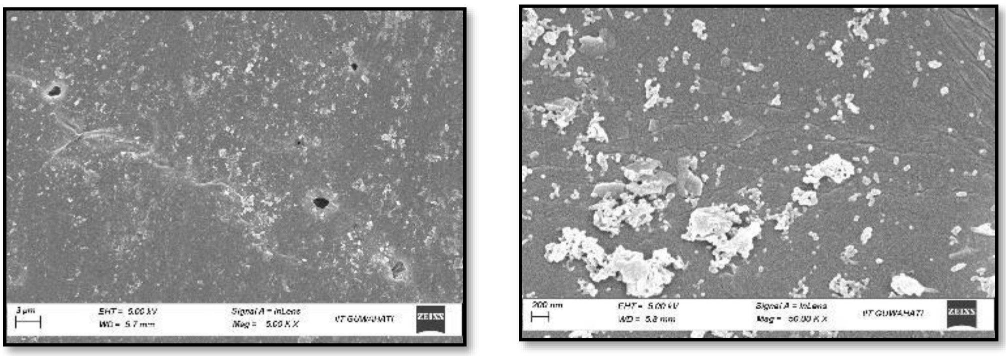


Fig 20 : FESEM image for Sample 03

(With nanomaterial, TiO-0.3%)

Fig 18 represents the FESEM image for the membrane Sample without Nanomaterials and Fig 19, 20 represents the FESEM image for the membrane incorporated with GO and TiO_2 nanoparticle respectively.

Calculation of porosity : Porosity of the polymeric membranes are calculated using the formula:

$$Overall\ porosity\ (\%) = \frac{w_w - w_d}{A\ X\ \delta \times \rho_\omega}$$

Here, the calculation of thickness of the membrane sample (δ) is discussed in (4.1.2).

TABLE 08: Calculation of porosity for different polymeric membranes

Sample No.	Polymer Concentration (w/w)	Nano Concentration (w/w)	Porosity (%)
01	14%	0%	45%
02	14%	0.3% Graphene Oxide	38%
03	14%	0.3% Titanium Oxide	35%

- By studying the images visually (Fig18,19), as well as interpreting the Porosity Data, we could conclude that, with the addition of Graphene Oxide nanoparticles, the pores obtained were almost similar in size with better pore distribution, with only a minor reduction in the overall membrane porosity. Similar trend is observed for the FESEM images of the membranes incorporated with Titanium Oxide Nanoparticles (Fig20), when compared to membrane without nanoparticle (Fig 18). Thus, this section provides us with the information that the synthesized membranes are porous and can be used for filtration or any seperation process , that will be discussed in the next sections.

- Moreover, from Fig (20), which represents the membrane incorporated with Titanium Oxide, when magnified up to 50000 times, providing magnification in the range of nanometres, it can be observed how the nanomaterials were dispersed within the membrane surface leading to a rougher surface. In this condition a dense and hydrophilic surface will form, which can result in lower foulants deposition on the membrane surface. The presence of nanoparticles in the membrane surface happens during the phase inversion process. The hydrophilic nanoparticles migrate spontaneously to the membrane / water interphase to decrease the interface energy.

4.1.2 Thickness of the Polymeric Membranes

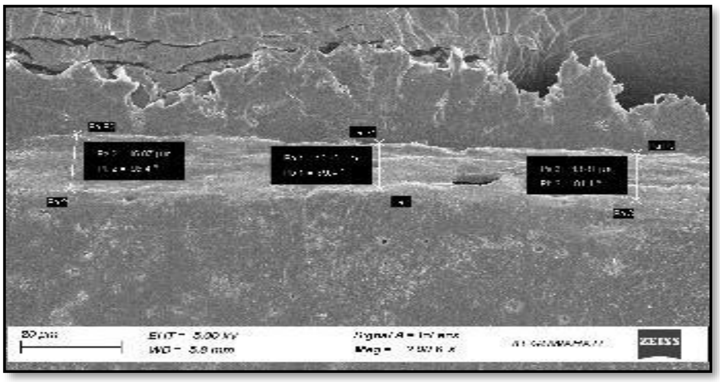


Fig 21 : FESEM cross sectional image for sample 03

For determining the thickness of our membranes, the Cross Sectional thickness of the Membrane incorporated with Titanium Oxide Nanoparticle was measured, using the Image-J software, from 3 different angles, as shown in fig 21, from which the Average thickness was found to be 15 micrometres. This thickness value was considered for the membranes of other compositions as well, since all the membranes were synthesized in the same Setup, keeping all the variables constant.

4.2 MEMBRANE HYDROPHILICITY USING CONTACT ANGLE EXPERIMENT

The results of sessile drop experiment performed at IASST (Institute of Advanced Study in Science and Technology) using a goniometer for two polymeric membrane samples (one with nanomaterial and another without nanomaterial) which shows the hydrophilic and hydrophobic nature of the polymeric membrane respectively are shown in Fig 22 and Fig 23.

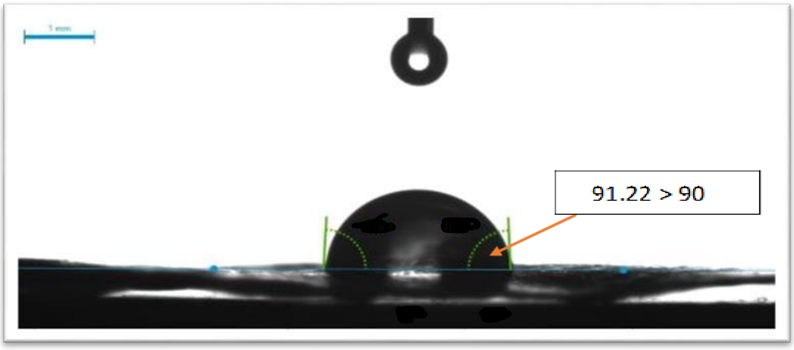


Fig 22: Sample 1 (without nanomaterial), here the contact angle is more than 90^0, therefore this sample shows a hydrophobic nature.

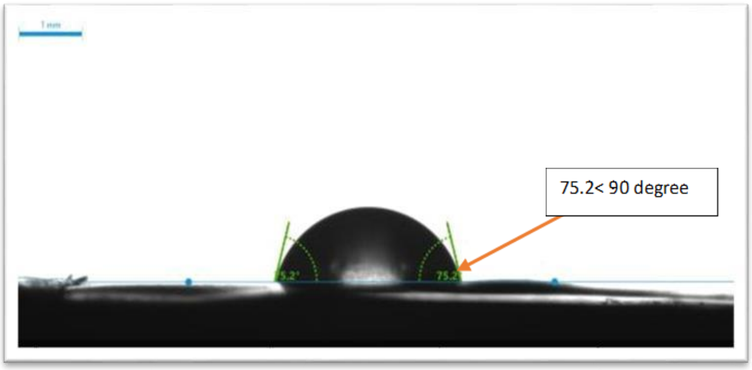


Fig 23: Sample 3 (with nanomaterial), here the contact angle is less than 90^0 , therefore this sample shows hydrophilic nature.

The results revealed that contact angle was significantly decreased from 91.22 ^{0} for unfilled polysulfone membrane to 75.2^0 for membrane filled with 0.3% TiO_2 nanoparticles. After incorporation of TiO_2 nanoparticles into the membrane matrix an improvement in membrane hydrophilicity was observed by reduction of contact angle.

The migration and presence of TiO_2 nanoparticles to the top surface of prepared membrane increases the hydrophilicity in comparison of unfilled polymeric membrane. Thus, higher surface hydrophilicity prevents foulant deposition due to compact water layer on to the membrane surface which can improve antifouling properties during filtration.

4.3 ACTIVATION OF THE POLYMERIC MEMBRANES

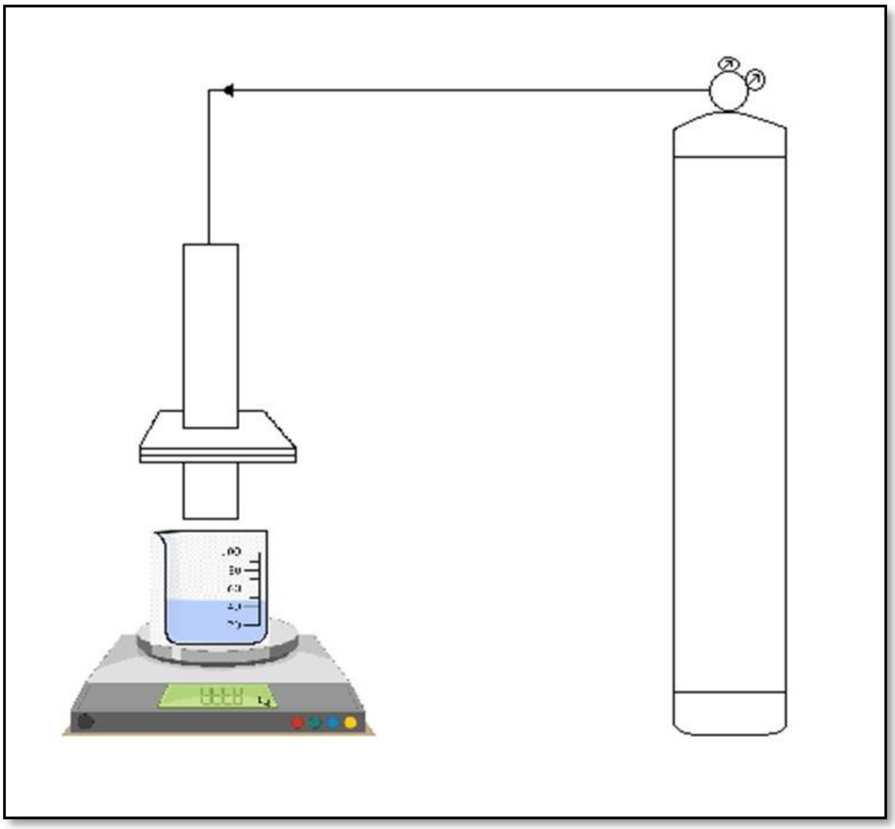


Fig 24: The Batch cell setup

TABLE 09: Time V/S Weight at a constant Pressure of 25 psi

With nanomaterial (TiO_2)

Time (in min)	Weight (in gram)
1	31.8
2	46.7
3	54.9
4	60.4
5	64.27
6	67.37
7	70.2
8	72.60
9	74.83
10	76.81

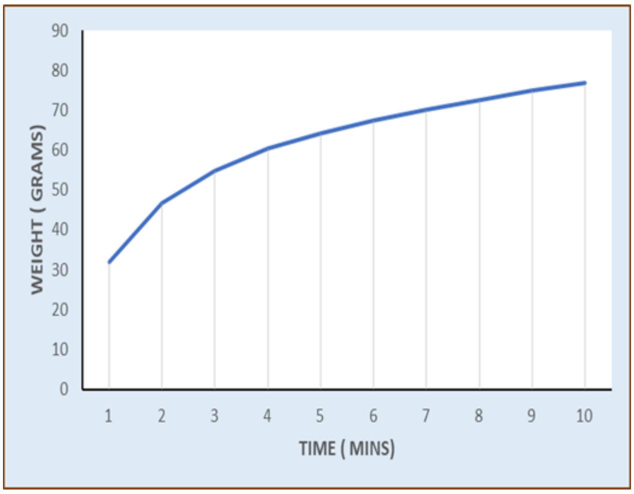


Fig 25: Constant pressure activation of sample 03

Table 10: Time V/s Weight (18psi)

Without Nanomaterial

Time (in min)	Weight (in gram)
1	43
2	65.7
3	80.5
4	92.8
5	103.14
6	112.57
7	120.5
8	127.8
9	134.7
10	141

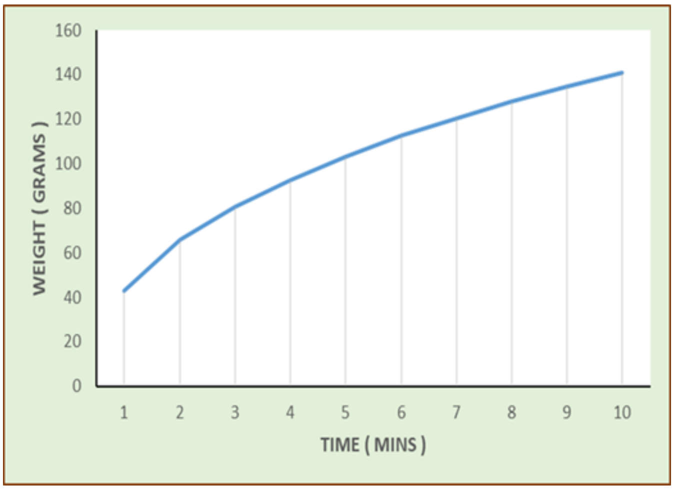


Fig 26: Constant pressure activation of sample 01

- From the data collected during the experiment, we have plot Time V/S Weight Curve for both the membranes at a constant pressure of 25 psi and 18 psi accordingly.

- As the weight differences gradually becomes equal with time, we get linear curves for both the membranes. Hence, we can infer that the membranes have now been activated.

4.3.1 Mechanical Strength of the Membranes

From the constant pressure activation process, it was observed that the sample 01 (purely polymeric membrane without nanoparticle) could resist a maximum pressure of up to 18 psi, after which the membrane gets ruptured. However, the membrane incorporated with 0.3% TiO_2 (Sample 03) could resist pressures exceeding 25 psi. Thus, the membrane shows increased Mechanical Strength on being incorporated by Nanomaterials.

4.4 DETERMINATION OF MEMBRANE PORE SIZE

For this experiment, we feed 100 ml of water from the top of the batch cell, whose flux is to be calculated. Inside the batch cell, we fit the membrane and pressurize the water through it, at 3 different pressures, as depicted in the tables below.

TABLE 11: Flux obtained for Nanocomposite Polymeric Membrane (TiO_2)

Pressure (Pa)	**Weight (10^{-3} kg)**	**Volume (10^{-6} m^3)**	**Flux (10^{-5} m^3/m^2s)**
86184.47	1.4	1.4	4.60
129276.7	2.1	2.1	6.90
172369	2.8	2.8	9.20

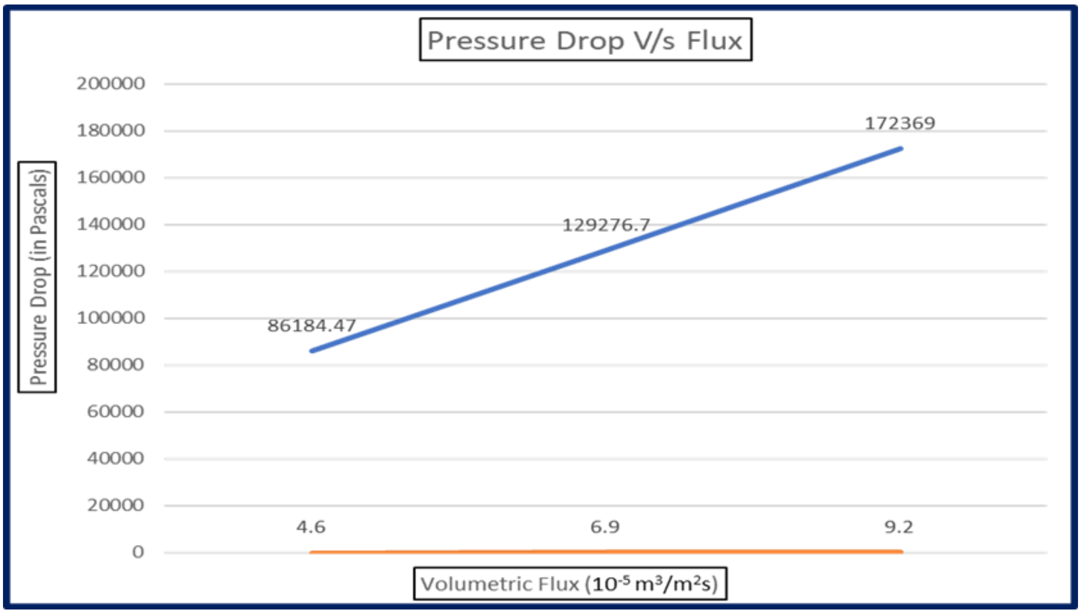


Fig 27 : Pressure drop v/s flux for sample 03

Here, The slope of the curve = 1837576308

From Darcy's Law, we know,

$$\Delta P = (\mu t/k)J$$

Thus,

Slope (m) of the ΔP V/s Flux Curve = μt/k

$$\boxed{\mu t/k = 1837576308}$$

Here,

Viscosity, $\mu = \mu_\omega = 10^{-3}$ Pa.s

Thickness, t = 15 μm (from section 4.1.2)

We get,

Permeability, k = 8.16 x 10^{-18}

Finally,

According to Hagen-Poiseuille's Equation,

$$k = (\varepsilon d_p^2)/32\tau = 8.16 \times 10^{-18} \text{ Henry/m}$$

Putting the known, Tortuosity, $\tau=1$

Porosity, $\varepsilon=35\%=0.35$ (From Table 08) ,

d_P = 27.3 nm

TABLE 12: Flux obtained for the polymeric membrane (without nanomaterial)

Pressure (Pa)	**Weight (10^{-3} kg)**	**Volume (10^{-6} m^3)**	**Flux (10^{-4} m^3/m^2s)**
86184.47	10	10	3.28
106868.0	12.8	12.8	4.20
127553	15.9	15.9	5.22

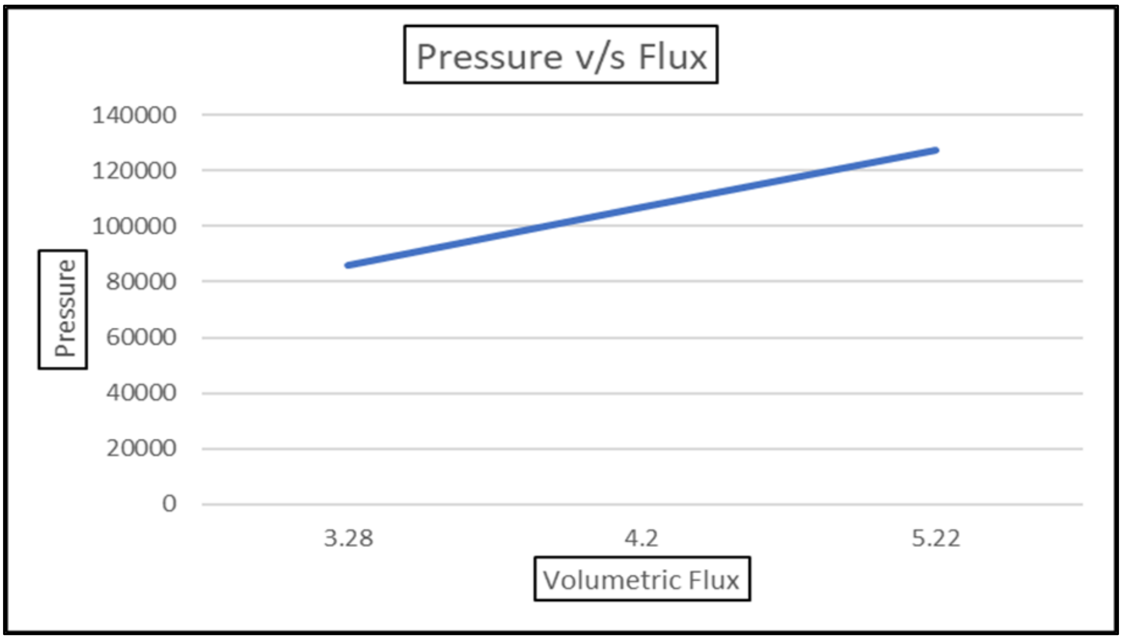


Fig 28 : Pressure drop v/s flux for sample 01

Here, from the graph, slope of the curve = 213807547

From **Darcy's Law**, we know,

$$\Delta P=(\mu t/k)J$$

Slope (m) of the ΔP V/s Flux Curve = $\mu t/k$

$\mu t/k$ = 213807547

Again,

$\mu=\mu_{\omega}=10^{-3}$ Pa.s

Thickness, $t = 15\ \mu m$

We get, **Permeability, k = 7.01 x 10^{-17}**

Finally,
According to **Hagen-Poiseuille's Equation**,

$k=(\varepsilon d_p^2)/32\tau$ = 7.01 x 10^{-17} Henry/m

Putting the known, $\tau=1$

Porosity, ε=45%=0.45

We get, **d_P = 27.3 nm**

INFERENCES

Polymeric Membrane with nanomaterial : 27.3 nm (Ultrafiltration range)

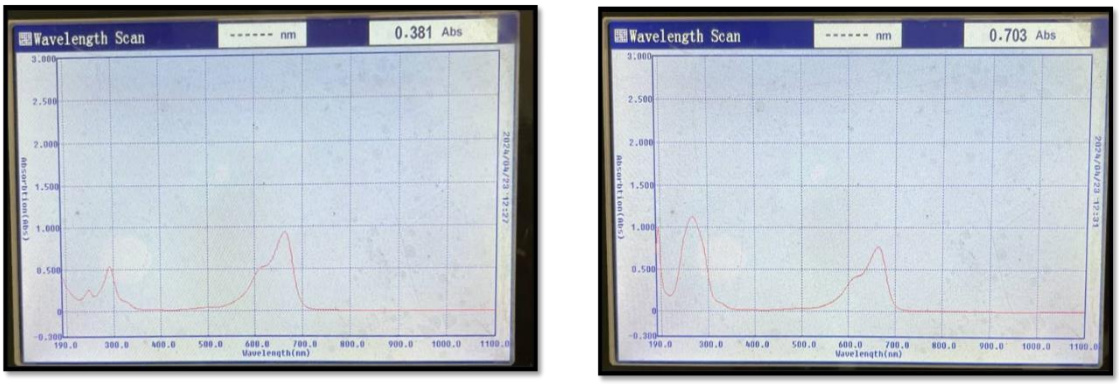
Polymeric Membrane without nanomaterial :70.6 nm (Transition between MF/UF)

- The result from this experiment supports our previous observation regarding porosity where we found that by incorporating nanomaterial the porosity decreases.
- By adding nanomaterial to the polymeric membrane, we can separate more finer particles.
- By incorporating nanomaterial to the polymeric membrane, the mechanical strength increases.

4.5 THE METHYLENE BLUE CALIBRATION CURVE

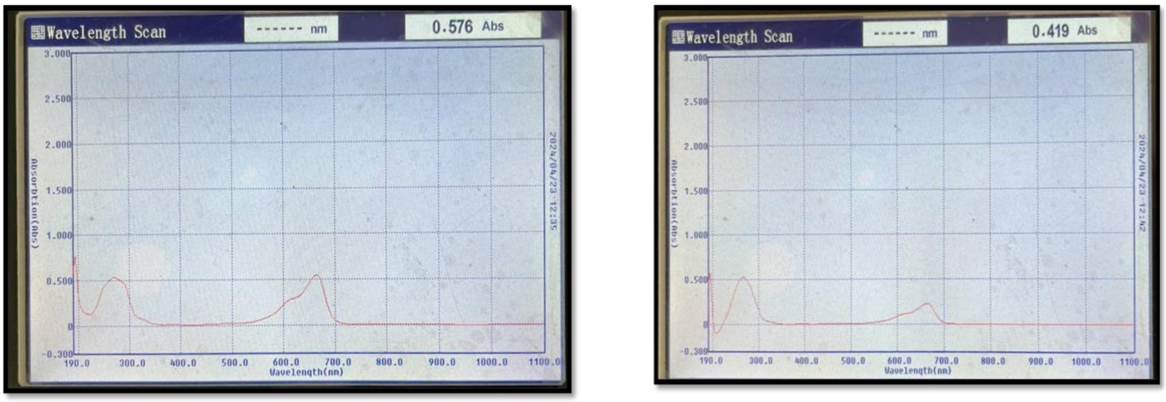
Methylene blue particles, a common industrial effluent, falls in the range of ultrafiltration, for which we have used the sample no. 03 to study the effectiveness of the membrane in separating methylene blue from a feed solution.

For the Construction of the Methylene Blue Calibration Curve, we have taken 6 solutions of Known Concentration, as mentioned in the table below, and plotted the concentration as a function of Absorbance, which is obtained from the UV-Vis Spectrometer, to obtain the Calibration Curve. The UV-Vis Images of certain known solutions are shown in the figures below.



(a) 5ppm

(b) 4ppm



(c)3ppm

(d) 1ppm

Fig 29 : UV Vis Spectroscopy diagrams of known samples (a),(b),(c) and (d)

TABLE 13: Concentration V/S Absorbance

Concentration (in ppm)	Absorbance
0.5	0.081
1.0	0.180
2.0	0.342
3.0	0.545
4.0	0.772
5.0	0.938

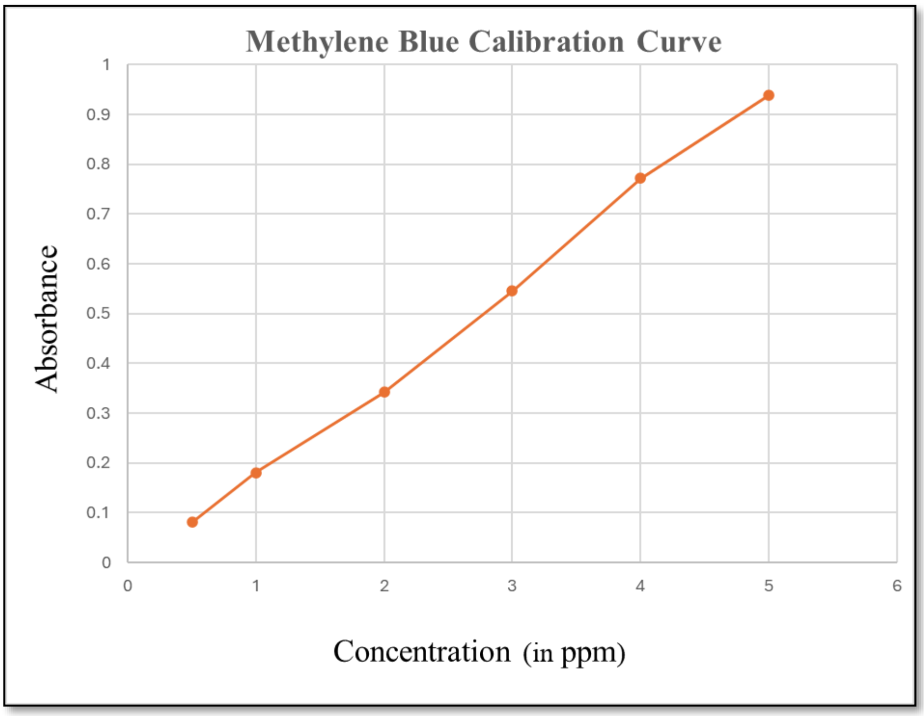


Fig 30: Calibration Curve of Methylene Blue

4.6 PERCENTAGE OF METHYLENE BLUE REJECTED BY THE PSf-TiO_2 NANO-COMPOSITE MEMBRANE

The feed solution, having concentration of 100 ppm is fed to the batch cell, and the experiment is carried out as mentioned in 3.5.7. The permeates were collected from the bottom of the setup after an equal interval of 4 minutes, and repeated for 3 independent permeate samples, to study the Reduction in the Removal Efficiency of MB from the Titanium Oxide Nano-Composite Membrane.

Permeate 1: Collected between 1st to 4th minute
Permeate 2: Collected between 5th to 8th minute
Permeate 3: Collected between 9th to 12th minute

TABLE 14 : Percentage of methylene blue separated in each permeate

Permeate No.	Dilution Factor	Absorbance of the Diluted Solution	Concentration of the Diluted Solution from Calibration Curve	Concentration of the Actual Permeate	Separation of MB Achieved from the Membrane
1	20	0.451	2.4 ppm	48 ppm	52 %
2	20	0.551	3.1 ppm	62 ppm	38%
3	20	0.706	3.7 ppm	74 ppm	26%

The Filtrate coming out from the bottom of the batch cell, cannot be sent directly to the UV-Vis Spectrometer to obtain it's Absorbance, as the Spike gives a very high absorbance, much higher than 1.0, which doesn't obey the Beer-Lambert's Law, stating that any solution showing absorbance greater than 1.0, will not follow linear behaviour as a function of its Concentration, and hence, it's concentration cannot be determined from the Calibration Curve.

Thus, the permeate coming out of the setup is first diluted, here, up to 20 times, and this diluted solution is sent to the UV-Vis Spectrometer to get the Absorbance Value. This value of absorbance is gives a corresponding Concentration of the Diluted Permeate, from the Calibration Curve, using which the Concentration of the actual permeate can be calculated by the formula given below:

(Conc. of the Permeate = Conc. of the Diluted Permeate X Dilution Factor)

4.7 REDUCTION IN THE METHYLENE BLUE REJECTION CAPACITY OF THE PSf-TiO_2 MEMBRANE

From Table 14, it was seen that the Membrane's ability to Remove MB from the Feed solution shows a gradual reduction from permeate 01 to permeate 03. This is due to the lack of availability of free active sites present at the membrane surface with the progress of time, which can absorb the Methylene Blue from the solution.

Another factor contributing to the slight reduction in removal capacity may be attributed to Pore Blockage due to agglomeration of the TiO_2 Nanoparticles or rupture of Active sites on the Membrane Surface.

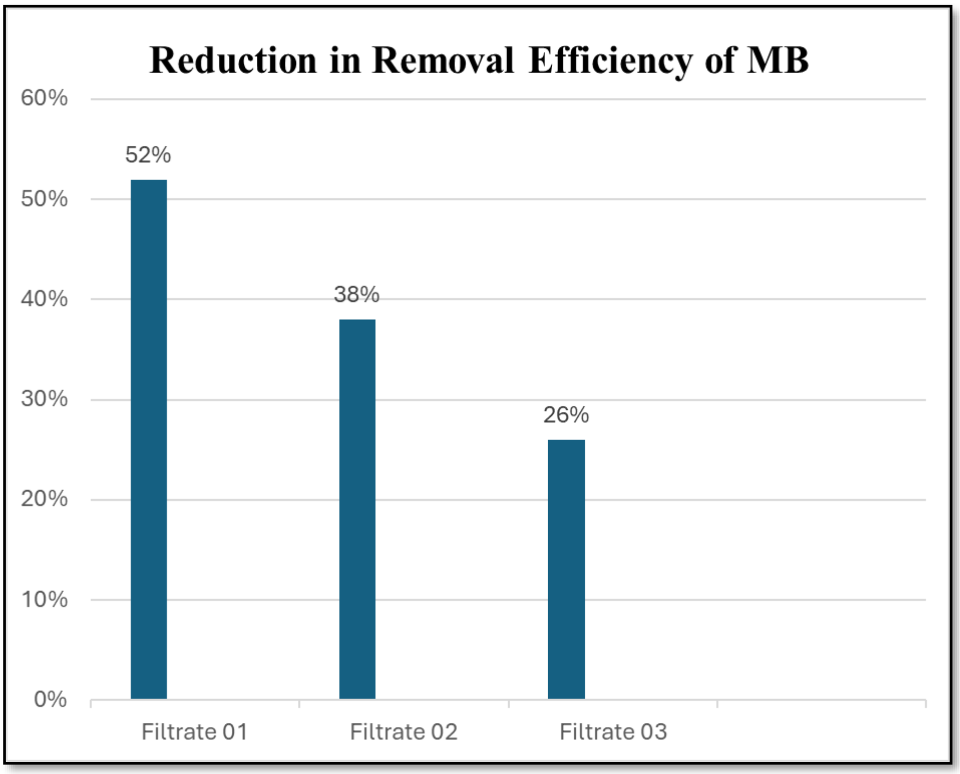


Fig 31 : Reduction in Removal efficiency of MB

CHAPTER 05

CONCLUSION

A key takeaway from the study is that the incorporation of nanomaterials into polymeric membranes has shown significantly enhanced membrane properties. The nanocomposite membranes exhibit improved hydrophilicity, mechanical strength, and selective permeability. Another valuable finding from the research is the verification of the study that the incorporation of Nanomaterials leads to reduction in the membrane porosity. Furthermore, this study focuses into the optimization of the Polymer-Nano concentration in the solution, through a systematic series of trials and errors, as detailed in the report. This discovery holds substantial implications for membrane fabrication processes, contributing valuable insights into achieving the desired membrane characteristics. In addition, the study discusses on Methylene Blue, a common industrial Effluent, about it's Calibration Curve, and the process of separation of MB from a feed using the PSf-TiO_2 membrane, by means of Ultrafiltration Process.

In conclusion, the study accomplishes its outlined objectives within the defined research scope. The primary goal of synthesizing Poly-nanocomposite membranes is effectively achieved through the testing of various combinations. Additionally, the objective of mitigating the hydrophobicity of polymeric membranes through the introduction of nanomaterials is also achieved, as verified by the results derived from the conducted contact angle experiment. Finally, the objective to remove MB from it's aqueous solution using the synthesized membranes was also achieved successfully.

This research represents a significant advancement in membrane technology, with potential applications in Dye removal from their solutions, such as Methylene Blue, and other associated fields of Purification. The enhanced Performance and versatility of these Polymeric Membranes, aided by the addition of Nanoparticles are expected to contribute substantially for addressing global Water Pollution from Industrial sources and related challenges

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