Mechanical Design of Prototype Pilot Scale Plant for Polymer Membrane Modules Manufacturing



By

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MASTER THESIS WORK

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"Patience is not an absence of action; rather it is timing. It waits on the right time to act."

Fulton J. Sheen

DEDICATION

I would like to dedicate this MS thesis to my loving Parents. It's their prayers, without them I would have never been able to complete my MS degree. It is their continuous love and support that keeps me going to achieve all my goals in life and to become a better person.

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LIST OF ABBREVIATION

SEM	Scanning Electron Microscopy	
PES	Polyethersulfone	
ATR-FTIR	Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy	
NMP	N-Methyl-2-Pyrrolidone	
MF	Microfiltration	
UF	Ultrafiltration	
RO	Reverse Osmosis	
NF	Nanofiltration	
UTM	Universal Testing Machine	
ISO	International Organization for Standardization	
CAD	Computer Aided Design	
3D	Three Dimensional	

ABSTRACT

Membranes were casted using non-woven polyester fabric as support material and using different concentrations of polyethersulfone as polymer material and NMP as solvent for the membranes. Immersion by precipitation technique was used to produce the pore morphology in the fabricated membranes. Scanning electron microscopy and optical profilometery were used to observe the morphologies and surface roughness of the fabricated membranes. It was observed that when the concentration of the polymer was increased the surface roughness of the membrane was increased as well. The tensile strength of the membranes increased with the increase of polymer in the casting solution. The pure water flux of the fabricated membranes was also tested at different pressures. The results indicated the dependence of flux on the surface roughness and morphology of the membranes. The flux observed to decrease with the increase of polymer weight percentage. The parameters used to prepare membranes were helpful in designing and fabricating the prototype pilot scale plant.

The project also involves to explore the design and development of pilot-scale prototype plant for flat sheet polymer membranes. This plant can use to make flat sheet membranes on pilot scale. It is a semi-automated machine, which can be operated by a single person. A mounted motor on the shaft is utilized to pull the support fabric. Precipitation time is a key factor for the formation of pores in the membrane. Controlled revolutions per minute of motor can provide enough time for precipitation of polymer in the bath tub. Rollers mounted on one another with calculated gap can ensure proper wetting of polymer on the support fabric. It occupies small space and can be used to make membranes on pilot scale.

1. PROGRAM OF RESEARCH

Present work is focused on the fabrication of the prototype pilot scale plant for polymeric membranes modules manufacturing through immersion by precipitation method.

The research project is divided into following phases:

1.1 Phase 1:

In the first phase, polyether sulfone membranes were made using immersion by precipitation method. The membranes were made by varying the concentrations of polyethersulfone and NMP was used as solvent, these were made in lab by manual method and fabricated membranes were then tested to verify the quality of the membranes. After the successful preparation of the membranes, the project entered the second phase.

1.2 Phase 2:

In the second phase of the project the membranes were thoroughly characterized using SEM, FTIR-ATR, mechanical testing, optical profilometry, Contact angle analysis and pure water flux testing by using a dead end filtration cell at different pressures. The obtained results were then correlated to the conditions used for the fabrication of the membranes.

1.3 Phase 3:

In the third phase of the project, first a conceptual model of the prototype plant was made then CAD design of prototype plant was produced and then fabrication of prototype pilot scale plant was explored and the conditions at which the membranes can be made in the lab, with the same conditions kept while designing and fabricating the prototype plant.

2. INTRODUCTION

The availability of clean drinking water is one of the biggest challenges today followed by treatment of waste water[1]. It is worth noting that the global fresh water demand from 1900 to 1995 increased by a factor of six, and this increase is almost double in figures than the increase in population[2]. From 1995 this trend increased and the lack of access to drinkable water and sanitation is a major source of disease mostly in developing countries[3].

As there is a continuous decline of clean water supplies, the research is now shifted to explore new techniques to purify water to meet the increasing demand of drinkable water supplies. For this purpose, polymeric membrane filtration systems are expected to play an important role to meet the demand of ever increasing need of clean water supplies, as population and industries are growing there is a need for the improvement in water usage efficiency. As technology is advancing day by day cost effective water filtration systems are resulting due technological improvements and water filtration system market based on polymeric materials is growing both in scale and market share and competing with conventional water filtration systems based on polymers to meet demand of increasing supplies of clean water will require reduction in operating cost and better performing membranes. Keeping in view of these requirements the researchers are now attracted towards the fabrication of new and better performing membranes. Polymer material itself determines the performance and properties of any polymeric membrane, for this purpose many polymeric materials are used to fabricate membranes with different performance and properties[4].

A membrane consists of thin layer of semi-permeable material separates different substances when a driving force is applied across the membrane[5]. Separation by membrane processes is increasing as the membranes are used to separate microorganism, particulates and organic materials, chemical substances alter the color, odor and taste of water and in addition they also react with the disinfectants and form disinfection byproducts and are needed to be separated[6]. The membrane processes usually used are microfiltration, ultrafiltration, Nano filtration and reverse osmosis processes[7]. The process of interest here is microfiltration membrane separation.

Microfiltration is defined as a membrane filtration system by using membranes having a pore size of at least 0.04 to 10 micrometer[8]. Microfiltration membranes separate materials such as silt, sand, clay and some microorganism including giardia lamblia, cryptosporidium and few more bacterial species. Microfiltration membranes are not good against viruses but when used with disinfectants they tend to control these in water[9, 10]. As the science is progressing there Is a growing emphasis to limit the usage of chemicals or their concentration that are used during treatment of water. Membrane separation physically removes the disease causing microorganism and limits the usage of chemical processes such as chlorination[11]. Microfiltration membranes are also used for the pretreatment to reverse osmosis and Nanofiltration to minimize the fouling of these systems. Both the mentioned systems are used for the desalination or removing of hardness from groundwater[12].

Membranes are usually manufactured from synthetic polymers while they can be prepared from other materials too but membranes for the filtration of drinking water are mostly made from the polymeric materials[13]. One of the reason for using polymeric material is cost effectiveness of the membranes because the polymeric material is less expensive than the other materials[14, 15]. Mechanical properties of the membranes are also important as membrane with greater strength can withstand large trans-membrane pressure allowing stability at high pressures[16]. Membranes can either be hydrophilic or hydrophobic, describing their ability to get wet and also their ability to resist fouling to some extent[17].

Both the microfiltration and ultrafiltration membranes can be fabricated from a wide range of materials such as polypropylene, cellulose acetate, polyethersulfone, polysulfone, poly-acrylonitrile and many other polymers[18, 19]. Each polymer has its own different properties such as degree of hydrophobicity, Ph. Tolerance, degree of hydrophilicity, flexibility and strength[15]. Membrane filters for water filtration are manufactured as hollow fiber or flat sheet and then these are used with different types of membrane modules.

In the current research work, polymeric membranes based on polyethersulfone were made. Polyethersulfone is one of the most important thermoplastic because of its relatively low cost, versatility, recyclability and good mechanical performance and it is used in many applications such as in the preparation of microporous membranes. Many techniques have been used to make membranes from polyethersulfone such as melt spinning, cold stretching and immersion by precipitation method. However, immersion by precipitation is a versatile method to prepare membranes from polyethersulfone. In this method polymer is dissolved in solvent by stirring for about 20 hours to make a homogenous mixture and de-mixing occurs in the coagulation bath during phase separation, solvent is removed and membrane is formed by the formation of porous structure[20].

The importance of the influence of concentration variation on properties of polymeric membranes, current work also focusses on the variation of polymer concentration on the characteristics, structure, morphology and performance of the PES membranes. Three different concentrations of the PES were used to fabricate microporous membranes. The effect of different concentrations such as 15, 18 and 21 percent of the polyethersulfone on the properties of the microporous membranes prepared via immersion by precipitation method were studied. To determine the performance and efficiency of the prepared membranes, the flux rate analysis was also performed with other characterization techniques. For this purpose, pure water flux was performed with different pressures.

The membrane market is composed of a few but big market portions such as sea water desalination, ultrapure water production, or hemodialysis, and a large number of small market portion in the food, chemical, or pharma sector, and in the treatment and recycling of industrial waste water[21].

It is very difficult to accurately forecast the future of membrane market. Since the demand for potable water of good quality is increasing at a fast pace and fresh water resources with good quality is decreasing worldwide, there is a need for energy efficient and processes with affordable prices for the production of high quality drinking water from surface, brackish and polluted water, the membrane market will continue to grow in this area. The major problem is fresh drinking water as it is decreasing day by day and in third world countries it is a major problem, as most of the population in these countries lack access to the drinkable water[2, 22]. Water purification system are available but these processes come with a price and it is difficult to sustain the system as membranes are needed to be changed after some time as fouling occurs and this part of the purification system is the most important one[23]. The growth of membrane market also depends on the development of new membranes with increased selectivity and higher fluxes and with other qualities such as thermal, chemical, mechanical strength and stability.

Membrane separation processes are very energy efficient than many of other separation techniques available and energy requirement is one of the cost determining factor, whereas investment and maintenance contribute towards overall process costs[24]. There are other

factors that should also be considered are pretreatment and post treatment techniques, the desired product quality and the feed mixture composition which has to be treated. For example, for water treatment in which membrane processes are utilized extensively, the cost of the process depends mainly on the feed water composition which might utilize different membrane processes[25]. For the purification of surface water and certain waste waters micro- and ultrafiltration can be used[6]. The energy requirement of these processes is very low. Both the microfiltration and ultrafiltration are in competition with other low energy consuming processes such as bio treatment or sand bed filtration.

The overall capacity of the operating plant also has an important role in total cost management. So cost reduction can be achieved by increasing the capacity of the plant[25]. Depending on the composition of feed water and the desired quality of product water, a combination of different processes can be appropriate. An example of this is ultrapure water for industrial usage, it uses a sequence of processes such as reverse osmosis combined with microfiltration to remove particle traces and ions are removed by ion exchange techniques. Microfiltration is used in combination with reverse osmosis for pre-treatment process[26].

Another important thing to mention is environmental impact of membrane for separation purposes and it is quite low, as no harmful chemicals are used in the processes that have to be discharged and there is no generation of heat either[27]. The only effluent that is collected after filtration is the concentrate and post treatments are required for this purpose as processing of the concentrate might become necessary[28]. For this purpose, chlorination can be applied so the sterility of the potable water can be ensured.

The membrane industry came into being with the development of membranes and membranes processes and this industry is growing steadily by 6.0 % every year[29]. The basic structure of membrane industry is heterogeneous as the size and approach of the companies towards market is quite different. As most of the companies are interested in the production of membrane products only[30]. The products they offer vary from hollow to flat sheet membranes with different properties for different applications. Whereas other companies manufacture complete membrane devices[31]. These companies buy the membrane products from other companies and provides filtration system to the customers, which sometime includes the design and operation of the plant[31]. These companies provide complete separation services to the customers which sometime includes a combination of different processes, in addition to membrane processes they can provide different chemical and

biological processes. Few companies provide system designs, membranes and operation of the plant. As the market of water supply systems and membrane is growing at a rapid pace and changing due to fluctuation of the industry because of mergers and acquisitions.

The membrane market consists of large market percentage that includes, sea water desalination, ultrapure and hemodialysis membranes and small market percentage of food, pharma and chemical and treatment of industrial effluent[32]. The large market for the above mentioned applications is dominated by small number of big companies. Although there are small companies working actively in the market providing services such as waste water treatment and services to the food, pharma and chemical industries.

Most of the companies which produces membrane also produce the modules for membranes. Flat sheet membrane is mostly used in the spiral wound modules and are mainly used for water purification and desalination[33, 34]. These modules have large membrane area per unit volume and these are used in most of the application for pretreatment[35]. The material and fabrication process the companies used to make membranes and modules are different. Some membrane producers make asymmetric membranes for ultrafiltration and microfiltration from polysulfone and polyethersulfone, polyvinyl chloride[36]. While other are making composite membranes. Big companies that provide whole water system for surface water treatment which includes membranes, modules and system, for them the membrane is a commercially available item and they utilize membrane by acquiring small and medium sized membrane manufacturers.

It is a well-known fact that today world faces a lack of fresh and clean water and more than a billion people do not have access to safe drinking water and about millions of people die each year due to drinking unsafe water because of the diseases transmitted by drinking this water[23]. Many other are suffering due to contamination, intestinal infections by parasites and diarrhea caused by waterborne microorganisms[37, 38] and these are becoming leading causes of malnutrition owing to improper digestion by people who are suffering by drinking this water[39] [3].

Present project also aim to explore the fabrication of a prototype pilot scale plant to produce membranes on industrial scale and to provide safe, clean drinking water to under privileged people who cannot afford expensive water filtration systems. The main focus of the research is to explore the fabrication of a indigenously designed semi-automatic plant to manufacture polymeric membranes for water filtration systems.

2.1 Role of Polymeric Materials and Membranes

One of the greatest challenges which is being faced by all of us in the 21st Century, the challenge is the provision of sustainable supplies of clean water at affordable costs [15]. In order to overcome this challenge, membrane technology based on polymeric materials is widely expected to provide breakthrough among the various technologies due to its comparative advantages in terms of energy efficiency. Although, extensive work is carried out to develop and improve membrane technology based on polymeric materials, but better membranes needed to be made with high flux, more selectivity, resistant to fouling, and to the chemicals, such as chlorine[4]. The research work carried out in this thesis aim to contribute to the technology of making polymer membranes at large scale in Pakistan to overcome various existing deficiencies present in the current membranes and thus provide opportunities to resolve them via employment of indigenously designed pilot scale plant to manufacture and utilize polymeric membranes for drinking water purposes.

In view of above, it has been widely realized that the clean water reservoirs are declining in Pakistan and in other developing countries and the situation will become worse and to overcome this situation cost effective water filtration membrane should be made to overcome this crisis. More than 70% of earth is covered with water but the quality of water is not suitable for human consumption. Thus, indigenous technology for water purification is essential to meet present and future supply of drinkable water. Polymeric membranes are used to purify water by several ways. As membrane processes utilize less energy this technology is more favorable than other technologies. This technology can be used to purify additional quantity of water by using less energy resources. It is an opportunity for polymer membrane technologists to provide pure water for the consumption of human. To meet this challenge cost effective membrane production is necessary. Membranes used for different purposes are made from polymers, therefore, future research and development must need to look into the employment of various polymeric materials.

2.2 Aim and Scope of Study

The current thesis is directed towards the fabrication of indigenously made polymeric membranes that can be used for water purification. The project is aimed to fabricate a prototype pilot scale plant which can then be used to make membranes for water purification and it is based on immersion by precipitation method for manufacturing polymeric membranes modules. Solvent variation, co-polymers and different polymeric membranes casting solutions using immersion by precipitation method can be used to make membrane on prototype plant.

By considering the parameters which influence the performance and properties of membranes it would be of great importance to investigate their contribution to control the characteristics of these newly developed membranes on the plant.

Cost effective membranes can be made by using technological improvements, market share and scale of membranes for water filtration have grown steadily. Due to such factors, membranes for water filtration applications are now competing economically with conventional water filtration techniques. The research work also aims to signify the potential of usefulness of polymer membranes for achieving water sustainability in the future. The water usage efficiency is important due to increase in population and industrialization on a global scale. The widespread potential which is available via membrane separations processes has sufficient scales to realize global sustainability because of the substantial reductions in operating cost. This project thus aims to highlight on these important issues and contribute towards the manufacturing of membranes for water purification for smaller communities in Pakistan and third world countries.

3. LITERATURE REVIEW

Membranes for water processes are used for the purification of drinking water and sewage water a well. Membranes for filtration are used extensively for the treatment process as these membranes increase the reuse of water both for industrial use and for the domestic se as well. The membrane technology is used in many industries namely the water supply for domestic use, for industrial processing, agriculture industry and many more.

3.1 Membrane Basics:

Membranes can be used with different operations for the treatment of water for drinking purposes. Few of them are briefly described here.

Membrane Separation as a Unit Operation

In the figure a flow diagram explains the membrane based water treatment process as a unit operation. There are two steps: In the first step there is a membrane pretreatment unit which removes the particulates and in the second step there is a reverse osmosis unit for the removal of macromolecules. There are other steps too such as addition of chlorine, to control PH, dechlorination. The membrane for research is pressure driven, in which a pressure is applied to produce the purified end product. [15].



Figure 1: Water purification unit operation flow diagram [14].

3.2 Two Membrane Transport Mechanisms:

In this technique there are two mechanisms: In the figure both mechanisms are shown a) pore flow and b) solution-diffusion.

In the first step pore flow filtration is shown. In the pore flow mechanism, the filtration is done through a sieving action of the membrane. The case in which the pore size is small such as in Nano filtration membranes, a low rejection of high valiant ions is caused by polymer surface charge[12, 13].



Figure 2: Transport mechanisms through the pores of membranes[14].

3.3 Filtration in Porous Membranes and Processes:

3.3.1 Filtration in Porous Membranes:

Both microfiltration and ultrafiltration membranes are pore flow membranes and Nano filtration membranes is a combination of both solution-diffusion and pore-flow mechanism. The figure shows the abilities of these membranes to filter different types of particles. The filtration membranes may have variable structure and pore size throughout the membrane thickness. The asymmetric membranes have thin layer with fine pores and these are also called screen filters because the filtration of particles occur at the membrane surface having a very thin and selective layer. In these membranes the filtration of large particles occurs at the surface and the in depth filter capture the solute particles by the action of adsorption and diffusion.



Figure 3: Types of membranes with their filtration capability[14].

3.3.2 Processes Used for Pore Flow Membranes

The porous membranes were made by making a concentrated solution of polymer in the solvent followed by adsorption in a liquid bath, which may consist of water or a mixture of water with solvent, in liquid bath the polymer is not miscible but the solvent is miscible. Other methods include solvent evaporation; water vapor capture from humid atmosphere can also be used instead of immersion in bath. These methods were developed by Pinnau and Koros[40].

3.3.2.1 Dry-Jet Wet Spinning

In this procedure, the dope solution is casted on a support fabric to provide support to the casted film. The hollow fiber membranes are casted with an evaporation step known as dry jet wet spinning process[41].

3.3.2.2 Loeb-Sourirajan Process

Loeb-Sourirajan process is one of the most important methods used to fabricate membranes. In this process, water is used as non-solvent in the coagulation bath and this process was introduced to fabricate cellulose acetate reverse osmosis membranes. As this process can be better controlled it is mostly used for fabricating microfiltration and ultrafiltration membranes[42].

3.3.2.3 Thermally Induced Phase Separation Technique

Among various methods to prepare membranes, this technique is one of the most convenient method to fabricate membranes having good characteristics. This method is similar to phase inversion method but there is a difference in this method as this method uses decrease of temperature for coagulation of polymer rather than non-solvent. A polymer cast solution is prepared and is then casted on a support material, after casting one side of the casted film is cooled to start the phase separation process and rest of the membrane is cooled slowly so the porous structure is formed as the cooling propagates. Depending upon the polymer used, the prepared membrane may be anisotropic or isotropic[43].

3.3.2.4 Materials Used for Polymer Membranes

Different types of polymers are used to prepare the membranes. Typically, polytetrafluoroethylene, polyvinylidene fluoride, polypropylene and polyethersulfone are used to prepare the membranes. Among these polymers polytetrafluoroethylene has more hydrophobicity, oxidation resistance, thermal and chemical stability, it is better than the other three polymers. But due to its high cost it is less used than the other mentioned polymers. polyvinylidene fluoride has low surface energy and good physical characteristics. Because of these properties it is mainly used to prepare hollow fiber membrane.

PP has hydrophobicity, thermal and chemical stability. Polyethersulfone is one of the most commonly used polymer for membrane fabrication because of its thermal, chemical, hydrophobic nature and mechanical properties. It is cheaper than the other mentioned polymers, that is why it is widely used to prepare commercial membranes[44, 45].

3.4 Criteria and Applications of Pore-Flow Membranes

In order to develop and use pore-flow type membranes, there is a need to meet certain criteria. Few of the important criteria are summarized below: -

Applications criteria	Attributes of Design	
Manufacturing Cost: Should be	Durability	
affordable	Stability: Robust; Mechanical; Thermal	
• Lifetimes: Long with durability	Resistance to Chemicals:	
characteristics	• Membrane Porosity: High	
• Permeate throughout: High	• Fouling: High Resistance	
	Surface chemistries: hydrophilic or	
	hydrophobic.	
Higher selectivity:	Possibility of flexibility for selecting surface	
Engineering of surface chemistries	properties.	
Pore size control		
Overall Cost: On the lower side.	Materials for membranes: Must be cost	
	effective.	
	Processing should be minimum.	

Table 1: Criteria of pore flow membranes.

There are numerous sectors which use membranes. Typical examples include:

- Pharmaceuticals and microelectronics industries are widely using micro filters for several decades[15]. Main purpose to use highly purified water for these important industries.
- Food and beverage industries are using MF and UF membranes. Microfiltration membranes are used to filter out bacterial and yeast cells[15].
- Most Nano filtration membranes are used to clean the already purified water to remove low levels of contaminants from the water, also by removing ions such as sulfates from municipal water to make it soft[46].
- In industries ultrafiltration membranes are used to remove oil or water emulsions for the recovery of process water.
- Oil and water emulsions are used for lubricating and cooling in machining operations, ultrafiltration membranes are used to filter out oil from water afterwards[46].

4. MATERIAL AND METHODS

The polymer and solvent used to prepare membranes were used as received. The membranes were fabricated from polyethersulfone (PES). NMP N-Methyl-2-pyrrolidone (Sigma-Aldrich) was used as a solvent. To support the membrane, non-woven polyester fabric Novatexx 2471 provided by Freudenberg (Germany) was used as a support material. Distilled water was used in the coagulation bath for phase separation.

4.1 Fabrication of Membranes

The membranes were fabricated using immersion by precipitation procedure. Typical example of the procedure is described below and also shown schematically in Figure. In the first step, different concentrations of PES (15, 18 and 21 wt.%) were taken in capped vials and then NMP as solvent was added in them. The vials were placed on a hot plate and stirred for several hours to melt and homogenize the PES with NMP. After the formation of solution, the membranes were casted on dried non-woven polyester as support material with the help of doctor blade with controlled speed. This was followed by immersion of casted membranes were in distilled water for phase separation at room temperature. The prepared membranes were

then air dried for few minutes. This process resulted in the formation of homogeneous white colored flat sheet films.

Serial	Membranes	Polyether sulfone	Solvents
No.		Concentration (Wt %)	
1.	M1/PES 15 %	15 %	NMP
2.	M2/PES 18 %	18 %	NMP
3.	M3/PES 21 %	21 %	NMP

Table 2: composition of the prepared Membranes.



Figure 4: Schematic of the selective main steps adopted to fabricate non-woven polyester supported.

4.2 **PES membranes:**

4.2.1 Characterization of Membranes:

The properties and performance of the fabricated membranes were studied by various techniques.

4.2.1.1 Fourier transform infrared spectroscopy (FTIR) ATR

Fourier transform infrared (FTIR) ATR is the preferable method of the infrared spectroscopy. In this technique the infrared radiations are passed across the sample. The sample is faced with infrared radiations some radiations are absorbed by the sample while some of the radiations are transmitted across the sample. The spectrum is produced by this action which represent the molecular transmission and absorption. This spectrum act as a molecular finger print for the sample and two unique molecular structures cannot produce the similar infrared spectrum. This make FTIR a useful technique to characterize the unknown material on the basis of their molecular absorption. FTIR is basically a qualitative analysis of every unique material but the size of peaks in the spectrum is the direct indication of the material amount present. By the use of modern software algorithms FTIR can be used for quantitative analysis.

FTIR is useful and better when compared to old infrared spectroscopic analysis due to following reasons

- It is non-destructive approach
- Its measurements are precise which do not require external calibration
- It has ability of the increased scanning speed
- It has mechanically simple setup

The schematic layout of FTIR apparatus is shown in the figure[47]



Figure 5: schematic layout of FTIR apparatus

4.2.1.2 SEM and Optical Profilometery Analyses

The magnification or resolution of an optical microscope is very limited for extensive microscopic studies. It is not possible to resolve the things smaller than the wave-length of visible light. Electron with kinetic energy 1ev has wavelength associated is 1.23nm approximately. So, it is possible to resolve things with in Nano-meter range using electrons. SEM is mainly used for:

- To study the topographical features
- To observe the morphology
- Checking Phase distribution
- Measure the compositional differences
- Small scale defect measurements
- Elemental composition through energy dispersive X-ray Spectroscopy (EDX analysis)

Scanning electron microscope uses electron beam of energy ranging from 1 to 1000,000 eV of energy, incident on the sample. The interaction of electrons with the sample gives important information about the sample. Following are the main signal used in SEM:

- 1. Secondary Electrons
- 2. Back Scattered electrons
- 3. Characteristic X-ray
- 4. Augers electrons:



Figure 6: Signals used in SEM

4.2.1.2.1 Sample Preparation

JEOL-6940A, SEM was used to observe the structure and features of membrane fabricated along with topography of polyether sulfone. Membranes were cut into 0.25cm² samples and cleaned with filter paper to remove surface contaminations. The membranes were immersed in liquid nitrogen for approximately 60 seconds to freeze. After freezing, the membranes were broken into smaller fragments and dried in ambient conditions. The samples were Gold coated using JFC-1500 Ion sputtering unit. Gold coating helps to increase conduction and avoid the charge accumulation on the surface, during electronic bombardment. The specimens were then place on the stage and the chamber was locked. High vacuum is used during SEM analysis.

The layout of the SEM apparatus is shown in the figure below.



Figure 7: SEM Apparatus Layout

Non-contact Optical profilometery was performed on membrane samples using NOBOVEA PS50 to measure the surface roughness and thickness of the membranes.

The apparatus makes use of the wave nature of light that compares the path difference between the two surfaces one the reference the other the test. The apparatus is able to measure the form, roughness and the parts precision. The deviation and the angles surfaces can all be studied through the profilometer.



Figure 8: Optical Profilometer Apparatus Layout.

4.2.1.3 Mechanical testing

Mechanical testing was carried out of the membranes using the standard ASTM D882 to investigate the effect of concentration variation of polyether sulfone on its mechanical properties.

As per the standard, the gauge length of the samples was 2cm while the total sample length was 7cm. The width of the samples was kept as 1.1cm. The membranes were tested using the Universal Testing Machine Shimadzu with software Trapezium. The maximum load limit is 20kN. The strain rate was 3mm/sec.

Tensile properties are a great deal in characterizing and identifying any material. It may differ with thickness, the preparation procedure, speed, the grips and the manner in which the extension is carried out.

Any material that is to endure a working cycle, needs to be tested under this balance for the reason to know more about the capacity and capability of the material to withstand the amount of pressure, in this case, for membrane.

Readings of force vs stroke were measured until fracture of the membranes and a tensile strength graph was plotted.



Figure 9: Universal Testing Machine Layout

4.1.2.4 Contact angle

Contact angle were calculated to study the effect of concentration variation on PES membrane hydrophilicity. Research have shown that contact angle of PES membrane increased with the increase of concentration shows hydrophobic character[48].

The assumptions that are principally undertaken are following two:

1) The drop should show symmetry about an axis (central). This assumption crosses out the idea of viewing the drop at an angle. Since from all side it be equal.

2) Viscosity and inertial forces should not affect the drop only the forces shaping it should be the gravity acting upon it and the interfacial tension.

5µL drop of distilled was dropped on the membrane surface using a needle tip. A magnified image of the drop was taken by a digital camera. Static contact angle of the droplet was calculated using ImageJ LBADSA (low-bond Axisymmetric Drop Shape Analysis) software in degrees.

Three readings were taken on each membrane sample at different places and an average value of the results was used for the contact angle.

4.1.2.5 Flux Testing

A filtration cell was used to test the permeation characteristics of the fabricated membranes.



Figure 10: The filtration equipment used to test the permeation performance of the prepared membranes.

The performance of the prepared membranes was tested at different pressures for pure water flux study. For this purpose, filtration equipment was attached to a vacuum pump with adjustable pressure gauge to obtain desirable pressure to permeate the water through the prepared porous membranes. For each experiment, 20 ml of water was taken in the fritted glass funnel and then pressure was applied by the vacuum pump to permeate the water through the membrane placed on glass support base.

The fabricated membranes were subjected to the permeation experimentation for the water at variable pressures to measure water permeability. Flux was calculated by using the equation:

$$J = v/a.t$$

Where V is the volume of water, A is the effective membrane area and t is the permeation time.

5. RESULTS AND DISCUSSION



5.1 Scanning electron microscopy

Figure 11: SEM Images of membranes

Figure 11 shows the SEM images of membranes with surface and cross sectional morphology. The concentration variation has visible effect on the morphology of the membranes The membrane with low polymer concentration has more pores on the surface than the membrane with high concentration of polymer. The thermodynamics and kinetics of membrane preparation is different with different concentration of polymer. Rapid demixing in first membrane with 15% of polymer resulted in thin membrane surface hence more pores on surface and more flux. Whereas there is a delayed demixing as polymer concentration is increased resulting in thick surface less pores and less water flux.

5.2 FTIR (ATR)

The FTIR analysis of polyethersulfone was performed with Bruker Alpha FTIR Spectrometer in the range 500 - 4000 cm⁻¹. As in figure 12 transmittance peaks detected were in accordance with the transmittance peaks of PES as mentioned in the literature. The indexing of different bonds can also be seen in the below mentioned FTIR spectra.



Figure 12: FTIR spectra of membranes.

The transmittance peak at 1000 shows C-O group asymmetric stretch, whereas at 1150 sulfone group S=O symmetric stretch, at 1540 C=C group stretching and C-H group bending at 720. The aromatic functional groups found are same as mentioned in the literature for the polyethersulfone[49].

5.3 Optical Profilometery

Surface roughness of the fabricated membranes was measured according to the standard ISO 4287.

Membrane Composition	Surface Roughness Ra (nm)
15% PES	5350
18% PES	14834
21% PES	23930

Table 3: Surface Roughness of membranes (nm).



Figure 13: Comparison of surface roughness of membranes.

The results indicate that when the concentration of the polyethersulfone was increased the average surface roughness of the membranes also increased. The membrane with 15% polyethersulfone has smoother surface while the roughness increased as the concentration was increased. The surface which has more roughness is more prone to fouling than the smoother surface. The pure water flux of the membrane with smooth surface is more than the membrane with rough surface.



5.4 Mechanical Testing:

Figure 14: Tensile strength of PES membranes.

An average of 3 readings was taken for each membrane. Comparing the results with literature it was observed that there is an increase in the tensile strength. While testing, the support layer was left intact to the membrane. The main purpose was to test and see how much stress the membranes can endure [50].

The mentioned membranes have different polyethersulfone concentration and there is a difference in strength of the membranes. It is observed that 21% PES has the greatest strength and 15% has the lowest strength, so it can be deduced that with the increase of concentration of PES the tensile strength of the membranes increases.

5.5 Contact Angle:

Water contact angle was performed to study the hydrophilic or hydrophobic nature of the prepared membranes with NMP as solvent. Contact angle was performed by taking three readings at different spots on membranes and then mean of three readings were taken as the average contact angle to evaluate the nature of the membranes. From the results it can be observed that when the concentration of PES was lowest at 15 % the membrane tend to be hydrophilic and when the concentration was increased to 21% the membrane has hydrophobic nature [51].



Figure 15: Contact Angle of water on the prepared membranes.



Figure 16: Pure water flux of membranes.

The flux was performed to determine the permeation rate of water through the membranes. The contact angle and surface roughness of the membrane both had effect on the flux. Different pressures were used to observe the change in the flux rate.

As the concentration increased the pure water flux of the membranes decreased and it may be due to thick and dense surface of the membrane with the increase of the polymer concentration as the figure 16 shows the flux rate of three membranes. The membrane with 15% polymer has the highest flux and as the concentration increased the flux decreased, due to more surface thickness of the membrane M3/PES 21% [52].

6. Conceptual Design of prototype pilot scale plant

As there is almost no literature available for large scale manufacturing of polymeric modules for filtration purposes, that how the membranes filters are made, the technology behind the manufacturing is not disclosed. Membranes can be made in lab with different polymer solutions having different properties and by using different methods, these membranes can be used for the application but the problem still remains about the manufacturing of membranes on large scale to be used for filtration of water. The main purpose of the project was to develop a prototype plant for manufacturing membranes modules, so these can be used for the filtration purposes for small communities, because the clean water supply is becoming a major problem in third world countries.

After brainstorming few designs were made, but in the end the design which was based on immersion by precipitation method was considered for fabrication. The design of prototype plant was made with parameters necessary for immersion by precipitation method.

6.1 First paper design of the pilot plant

The first design was made on paper and it was aimed to fabricate a prototype pilot scale plant for polymeric membrane modules manufacturing, some of the dimensions of the plant were changed in the CAD design of the plant. The initial design was the concept design about different parameters of membrane manufacturing by immersion by precipitation method, the casting conditions, blade for desired thickness of membrane, coagulation bath for phase separation, dryer for drying of wet membranes and the same conditions studied during membrane fabrication in the lab.

6.2 Design Finalizing Factors

6.2.1 Material cost

Material cost in the project was approximately 25 thousand rupees, as one of the objective of the project was to develop a prototype plant in available resources. The approximate cost of the plant is for the first time development as few motors were tested for the rear shaft of the plant for driving the fabric across the plant. Material cost also include the electrical components for controlling shaft motor, also it includes labor and services that were

used outside the university. Most of the material used was second hand and recyclable material. After initial design the size of the plant was reduced to make it more compact and cost effective.

6.2.2 Availability of the material

Availability of the material was one of the most important aspect of the project because during the time of fabrication the availability of the material in the market was important, because considering those items that were not available in the local market would take a lot of time to complete the project. The cost of the new material varies among suppliers and also prices change very quickly on every fresh import. The components that were considered for the project were available in the local market, as most of the components were fabricated from reused material and bought from reused material shops.

6.2.3 Manufacturing cost

A main point of concern in manufacturing project is cost efficiency. One of the main advantages on working on this project was the availability of manufacturing resource center in the school, the machinery, tools, guidance and technical professionals were available to complete the project. A portion of research fund was saved by working in manufacturing resource center.

6.2.4 Manufacturing Time

Manufacturing time is important factor in manufacturing projects, lesser the manufacturing time lesser will be the machine cost and labor. Cutting, welding, machining and paint job was done in the first phase. The motor and its speed controller was designed later according to the manufactured plant requirements.

6.2.5 Running Cost

Another important matter is running cost of any machine; it is always preferable to keep the running cost of any machine as low as possible. The attached motor consumes very little electricity, and it is the only electrical component in the plant, therefore the plant is simple and economical in the long run.

6.2.6 Maintenance cost

Every machine requires maintenance after a period of time. This should be kept in mind while designing any machine as this is one of the important factor. Cheap maintenance but simple is always preferable. Reused printer rollers were preferred because they require less maintenance and can be replaced cheaply if required. Material other than printer roller may require special machining after a certain period of time and can increase maintenance cost of the machine.

6.2.7 Structural Integrity of machine

Structural integrity of machine is also important as it is related to number of components of machine and joints in machine. The model has more structural integrity.

6.2.8 Safety

It is one of the most important point and should be one number priority before designing any machine. Any machine that is designed and fabricated should be as much safe as possible. The model of the machine was very much safe.

6.3 Designing of prototype plant

The designing phase consist of the following steps.



Figure 17: Phases of design.

6.3.1 Preliminary Design

After designing the conceptual model, then comes the step of detail modelling of the prototype plant. An iterative method was used for modelling the prototype plant. An iterative method is a method which includes a sequence of approximate improving solutions for a problem are used to get the best possible solution and in this case the computer aided design model of prototype plant. The computer aided design model of prototype plant have two kind of components, those that were available in the market as finished parts and other parts which were designed and fabricated.

The design phase consists of following steps:

- a) First of all, the conceptual model and specifications of the prototype plant.
- b) Detailed computer aided design (CAD) model of prototype plant.
- c) List of all the components to be fabricated and bought from the market.
 Design specification and detailed computer aided design will be discussed with all the components with their brief description.

6.4 Manufacturing

The flow chart of the chapter



Figure 18: Flow Chart of the Chapter.

6.5 Orthographic Drawings

The first step was to make orthographic drawings from the 3D model. Few of the items used in the prototype plant were brought from the market while other were manufactured from the raw material. The parts which were bought ready made from the market are included in reference category, while other in raw material category.

The orthographic drawings category both reference and raw contains following information.

- 1. Name of the part.
- 2. Dimensions of the part.
- 3. Material of the part.

6.6 Process Planning

Material was purchased and process planning was completed by using the information provided by the orthographic drawings and floor shop availability. The key elements of good process plan are:

- 1. Name of the part, material, number of parts used and total approximate time.
- 2. Machining description of manufacturing operation.
- 3. Shop to be used for the operation.
- 4. Machines for the specific operation.
- 5. Type of tools and size of tool.
- 6. Cutting speed, feed rate of machine used in the operation.

Process Planning and machine time calculations

The time required for a machining operation has following elements

- 1. Handling time.
- 2. Set up time.
- 3. Machining time.
- 4. Tear down time.
- 5. Down time.

Out of five, the four effective times were picked and these are shown in the following flow charts given below:



Figure 19: Process Planning Time Analysis.

6.7 List of tools and machine

It is important to know the list of tools and machines used during manufacturing, it also helps in reducing the setting time. It also gives information about the shop that the capacity of the shop is enough for producing the desired product or not.

The list of tools and machines used in the manufacturing of prototype plant are as follows.

Serial No.	Machines	Tools	Marking/measuring
			Tools
1.	Milling machine	End milling cutter	
2.	Lathe machine	Threading tool, parting, facing. (Tip made of carbide)	Vernier caliper, Chalk, ruler, center
3.	Bench drill machine	5,8,10 mm drill bits	punch, scriber.
4.	Grinding Wheel		
5.	Abrasive saw	Cutting disk	
6.	Arc wedding	E1013 Electrodes	
7.		Filer tool (Round, flat).	

6.8 Assembly Design

Design for manufacturing rules were followed in the available resources. Tables are base frames for other components as other components are attached in such a way that assembly is easy and it saves time. The table was designed in such a way that there are few parts in the assembly, motor and shaft are assembled together, no need to assemble them separately. Machining and assembly time was reduced by using less parts in the plant. There was another consideration during the assembly phase designing to keep all the components inside the tables and tub to make the prototype plant much safer for people working near the plant.

7. Discussion

The aim of this research was to design a prototype plant for polymeric membrane modules manufacturing, in low budget, under available resources, and options for modifications in future. For designing a prototype plant immersion by precipitation method was selected and conceptual design was made. The main factors on which the design was finalized were the cost of the material, availability of the material, manufacturing cost, manufacturing time, structural integrity, and safety.

After the selection of the conceptual design, a computer aided design with specifications was developed. Detailed 3D design of every component involved in the prototype plant were made. Factor of safety was kept in mind and only electrical instrument with insulated wires was used and sharp metal edges were removed by filing tool. Manufacturing of the prototype plant was started with orthographic drawings of the parts used in plant. The orthographic drawing of the parts which were bought readymade from the market was also made. The parts manufacturing was distributed depending on the available shop in the resource center. The manufacturing resource center machinery and tools were used to manufacture the parts from the raw material. The manufacturing tasks were completed on daily basis, availability of machine and tools were checked on advance basis to confirm the next day task. The fabricated parts and readymade parts can be assembled by using the assembly design of the prototype plant and prototype plant can then be tested by making membranes on the plant.

8. Conclusions

The membranes fabricated were tested to study the effect of different concentration of the polymer on pure water flux, their strength and on the surface of the membranes. It was studied that when the concentration of the polymer was increased the contact angle become higher than the membrane with lower polymer weight percentage used and at the same time the membrane with low polymer concentration showed increased flux rate at different pressures as this is due to more porous structure of membrane with low concentration accompanied by decreased surface roughness. This research was aimed to study parameters for membrane manufacturing to fabricate a prototype pilot scale plant to produce polymeric membranes on large scale and to eliminate manual work, the proposed plan is to fabricate a unit which can work on the same principle by which the above mentioned membranes were fabricated i.e. by using the principle of immersion by precipitation method and to make membranes with different compositions which can enhance properties of membranes such as addition of additives in the dope solution and nanoparticles to fabricate membranes with antimicrobial activity, antifouling, increased flux and hydrophilicity.

Prototype pilot scale plant for polymeric membrane modules manufacturing was also explored. The membranes prepared in lab conditions can be replicated in the plant because same conditions were kept in mind while designing the prototype plant, the basic principle of membrane modules manufacturing is immersion by precipitation method, one of the technique of phase separation. This has the ability to make membranes from dope solutions having different polymeric concentrations with different solvents, addition of additives and nanoparticles can also be used in the dope solution. The controlled speed of the motor ensures enough time for fabric to immerse in the polymer solution tray with time needed in the precipitation bath tub for complete precipitation of membrane in the non-solvent.

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