

# DEVELOPMENT OF A POLYMER-MEMBRANE-BASED MODULE FOR THE SEPARATION OF CO<sub>2</sub> FROM N<sub>2</sub>

By

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## **ABSTRACT**

The main cause of CO<sub>2</sub> is the burning of fossil fuels. A few examples of indirect human-induced effects on forestry and other land uses that might produce CO<sub>2</sub> are deforestation, terracing for agriculture, and soil degradation. Vehicles are mobile sources of CO<sub>2</sub> emission, although the chemical and petrochemical industries are permanent sources as well. These are the primary reasons behind Earth's global warming. Natural gas must be purified of these pollutants before being used. Today, significant amounts of energy are used for separation and purification processes, most inefficient primarily due to thermodynamic limitations. Membrane-based technologies, as an alternative, have grown in popularity because they can combine reliable operation with molecularly selective separation, low maintenance, economic effectiveness, and energy-efficient operation. The separation of the necessary gas component from the gaseous mixture depends on the selectivity and permeability of the membrane. In accordance with our plan, scaled-up assisted transport membranes for nitrogen and carbon dioxide separation will be produced as spiral-wound modules. The area of the membrane in each module was appropriately expanded using a multi-leave wounding technique that used a carrier sheet, and the assembly processes had been thoroughly explained. Gas permeation technology was utilized to isolate nitrogen from the essential gas components (CO<sub>2</sub>). The combination of two methods, the phase inversion method, and the solvent evaporation method was used. As a result, the gas-separating spiral wound module was created by applying the Gas Permeation technology to optimize its operating conditions and efficiency. It was concluded that the permeability and selectivity of a module can be increased further if leakage of the module can be controlled.

**Keywords:** selectivity, permeability, spiral-wound membrane module, gas permeation technique

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## **DEDICATION**

This entire study is dedicated to our loving parents, who have continually offered their moral and financial support as well as been our sources of motivation and strength when we felt like giving up. To all of our respected teachers who provided their assistance and support so that we could complete our studies. Finally, we would like to express our thankfulness to Allah Almighty for giving us skills, direction, strength, and mental power.

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# **1 CHAPTER 1. INTRODUCTION**

## **1.1 EFFECT OF CO<sub>2</sub> EMISSION**

A rise in the concentration of greenhouse gases is the primary factor contributing to the environmental issue of the twenty-first century, global warming[1]. According to projections, by the year 2030, the 95 trillion cubic feet of natural gas utilized globally in 2003 will have increased to 182 trillion cubic feet. (2012) Yeo et al. The burning of petroleum/fuel accounts for 17% of the CO<sub>2</sub> emitted by automobile combustion engines (fossil fuels). The UN called the Earth Summit in Brazil in June 1992. In order to alleviate the amount of greenhouse gases in the atmosphere, the agreement was made to hasten research and development for CO<sub>2</sub> capture, including the creation of membranes [2]. Given the daily increase in demand, natural gas is one of the most noteworthy fossil energies[3]. Energy Information Administration reported that 10 percent power sector in the entire world depends upon natural gas and its consumption is increasing by 4% annually[4]. Natural gas comprises different chemical species such as carbon dioxide, methane, ethane, N<sub>2</sub>, water vapors, butane, H<sub>2</sub>S, etc. but the acidic gases such as H<sub>2</sub>S, CO<sub>2</sub>, and water vapors are impurities as these gases raised safety and health concerns which must be removed [5, 6].

## **1.2 SEPARATION PROCESSES**

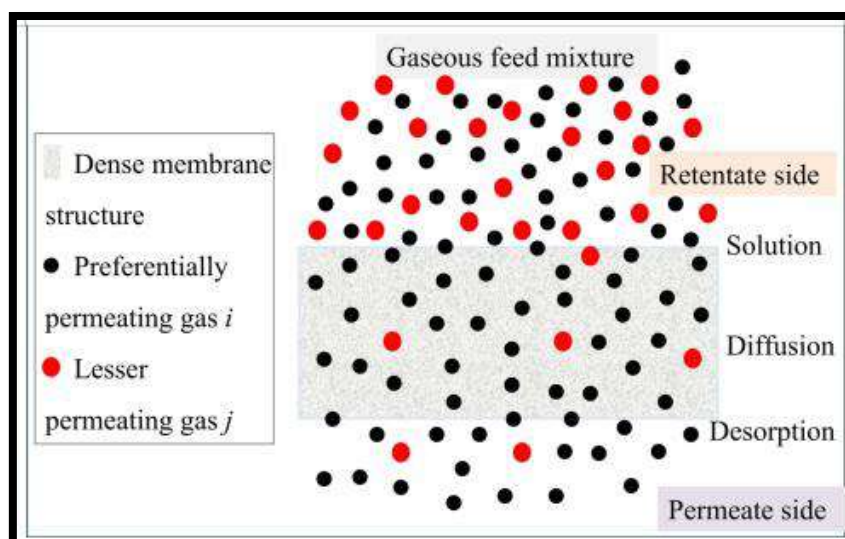
The process of separating components from a mixture and transforming them into various products is known as the separation process. Adsorption, crystallization absorption, distillation, solvent extraction, stripping, and membrane separation are a few of the several separation processes. Among these separation processes, it is possible to achieve many qualities including cost-effectiveness, energy efficiency, environmental friendliness, and excellent separation efficiency. Gas separation may be accomplished by membrane separation, which is economical, simple to install, and energy-efficient. Because of their mechanical strength and flexibility, polymeric membranes dominate separation procedures for instance flue gas separation, landfill gas recovery, air separation, natural gas sweetening, and hydrogen recovery [2, 7].

In a membrane involved segregation procedure, two majority phases are required, and the membrane acts as the 3rd phase in physically separating the two phases. The two phases of the input gas that are separated are permeate and retentive, which the membrane

carries through and holds. Operating parameters and membrane components control the flow of gas between these phases [2].

Several processes can support the transportation of gases via membranes, depending on the kind of gas combination and the type of membrane. The three most significant processes are Knudsen diffusion, molecular sieving, and solution diffusion. What causes penetration is the difference in chemical potential beyond the membrane [8].

Molecular sieving or molecular weight is used to separate gases in porous membranes (Knudsen diffusion). Membranes with molecular sieving mechanisms often have high production costs. When compared to other approaches, membranes based on the Knudson diffusion mechanism perform poorly in separation, making them unsuitable for commercialization in the industrial sector. Non-porous membranes are hence the foundation for the vast majority of gas separation processes. As gases travel through thick or impermeable polymeric membranes, the solution-diffusion process regulates how much of the gas is released. This theory states that the gas molecules absorb into the membranes at their upper stream side in the first stage, diffuse through the polymer matrix in the second stage in line with Fick's Law, and then desorb on the downside stream of the film in the 3rd stage. The components separate as a result of one of the permeating components' preferred sorption and faster diffusion [9].



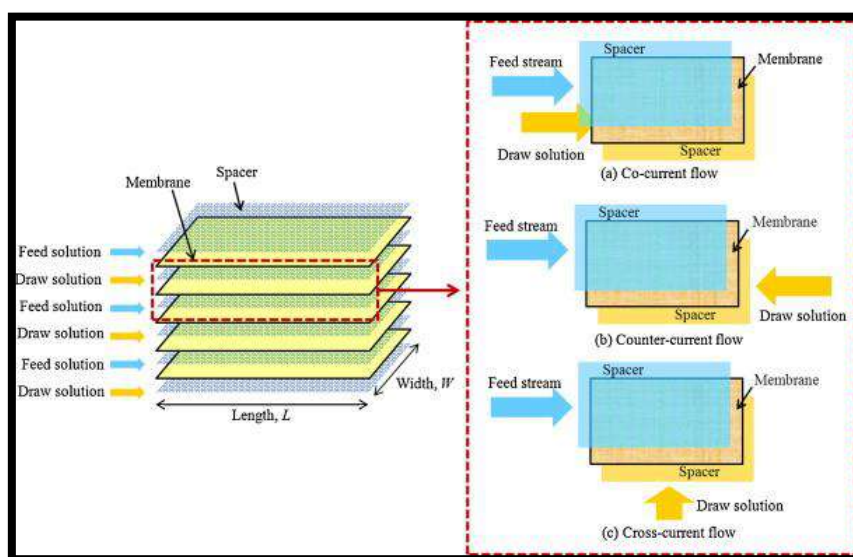
**Figure 1-1** Gas separation by using solution-diffusion mechanism [8]

### 1.3 TYPES OF MODULES

Membrane modules: On a commercial scale, industrial units need a lot of membrane area to perform separation operations effectively. The components or whole system necessary for installing these membranes in plants prior to the separation process. Membrane modules are the names for these housings for membranes. The popular gas separation membranes are made up of polymers and constructed in various module designs [10]. Numerous studies examine the flow dynamics in various configurations of modules used for gas separation [11]. SW modules and hollow fiber modules were recently discovered to be the most widely used commercially available modules [11, 12].

#### 1.3.1 Plate and Frame Membrane Module

For the purpose of removing He (helium) from natural gas, Stern et al. presented a plate and frame module design [7]. That module can only be used for ultrafiltration and microfiltration in low pressure applications. Due to their higher price than the other modules, difficult production, and poor membrane area to volume ratio, these units can only be used in a restricted number of applications [12].



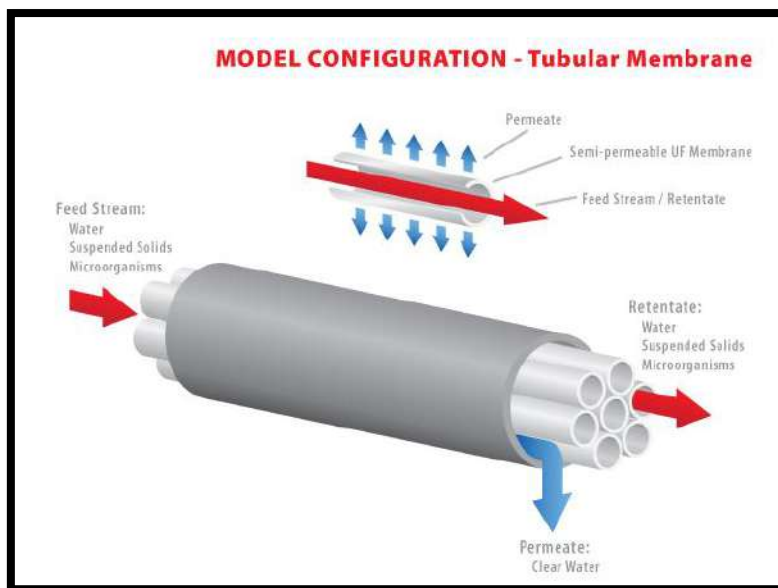
**Figure 1-2** Schematics of plate-and-frame module and possible flow guidelines of two solutions [11]

#### 1.3.2 Tubular Membrane Module

The execution of the procedure is largely reliant on on the membrane designs. The optimum membrane modules or configurations should have a variety of qualities, including a high membrane area to module volume ratio, ease of cleaning and cleaning

accessibility, increased turbulence, and affordability. However, none of these qualities can be attained simultaneously [13].

Numerous small-diameter porous-membrane tubes put inside a pressure vessel make up tubular modules. These modules offer easy manufacture, simple design, and fouling resistance [16]. Due to the extreme resistance to fouling provided by this design, these modules are only suitable for ultrafiltration operations [14].



**Figure 1-3** Schematic diagram of tubular membrane module [14]

### 1.3.3 Hollow Membrane Module

Another concept that offers dense packing and cheap manufacturing costs is hollow fiber modules. These modules are made of tiny, fine hollow fibers that offer dense packing. When feed into the shell is injected under pressure during the gas separation process, hollow fiber modules provide an issue with a high pressure drop within the bore side. Designing gas separation modules is primarily concerned with providing mechanical support for membranes, improving efficiency, and creating an outlet for permeates [10].

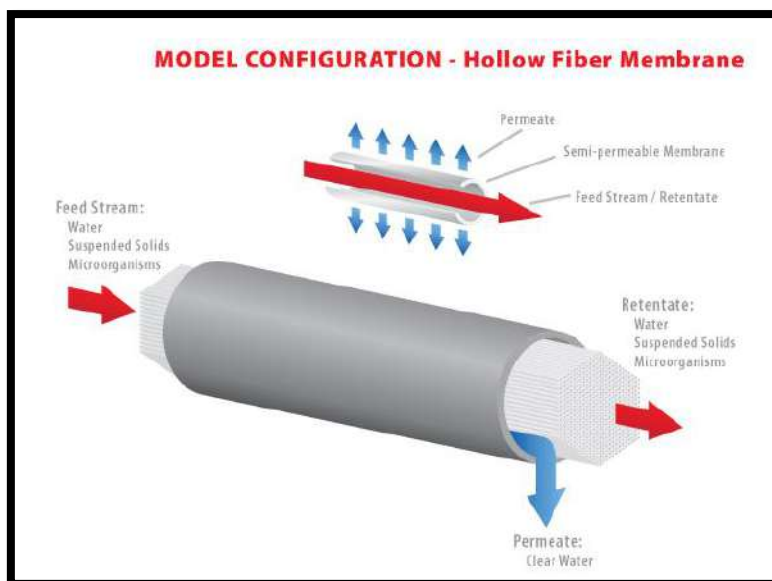


Figure 1-4 Hollow fiber membrane module [13]

### 1.3.4 Spiral Wound Membrane Module

Membrane is spirally looped around a perforated permeate collecting tube in the module. The membrane sheet in conjunction with the feed channel spacer creates turbulence and creates room for feed to flow uniformly [15]. The feed channel spacer and permeate spacer sheet are then joined, and the membrane is spiraled around the collecting tube as seen in figure 1-5. This design offers an adequate surface area to volume ratio and is easy to manufacture. RO and ultrafiltration both employ the wrappings module [12, 16].

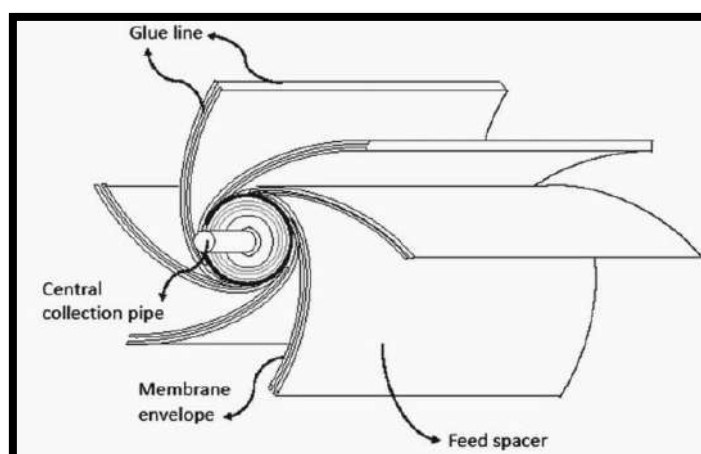


Figure 1-5 Multi-envelope SW module having 7 envelopes [5]

The membrane separation technique provides a number of advantages over the adsorption and absorption procedures for CO<sub>2</sub> capture in plants. For extensive gas separation, specific modules include hollow fiber, plate and frame, and spiral wound. Membrane wounds used the membrane techniques for gas separation effectively. Spiral wound design is appropriate for the treatment of natural gas containing 4–8 moles percent of N<sub>2</sub>, according to Lokhandwala et al. [17].

Designing a multi-envelop spiral wrapped module for effective CO<sub>2</sub> extraction from flue gases is the focus of our effort. This module was chosen because to its beneficial qualities, such as low manufacturing costs, high production rates (which produce strong turbulence), simpler maintenance, and high packing density. When a single-envelop module is swapped out for a multi-envelop module, the excessive pressure drop on the feed side is avoided. Hollow fiber modules are substituted with spiral wound modules because they offer greater resistance to fouling and regulate concentration polarization.

#### **1.4 UTILIZATION OF CAPTURED CO<sub>2</sub>**

By using carbon-containing fossil fuels sparingly, industrial plants may lower the quantity of CO<sub>2</sub> they produce. Utilizing the CO<sub>2</sub> that is collected from these industrial processes, which is a source of carbon, reduces the amount of greenhouse gases that are released into the environment. The CO<sub>2</sub> that is removed from fuel gases can be used to produce common compounds (like urea nitrogen fertilizer), carbon-based fuels, biofuels made from photosynthetic microorganisms, and other things. Therefore, CO<sub>2</sub> collection and its use are essential for lowering CO<sub>2</sub> emissions into the environment [15, 18].

#### **1.5 OUR AIMS AND OBJECTIVES**

Aims and objectives of this project include; Synthesis of polymer-based membranes for separating CO<sub>2</sub> and N<sub>2</sub>, insertion of prepared membranes into spiral-wound module, and designing, assembling and evaluation of module using Gas Permeation Setup.

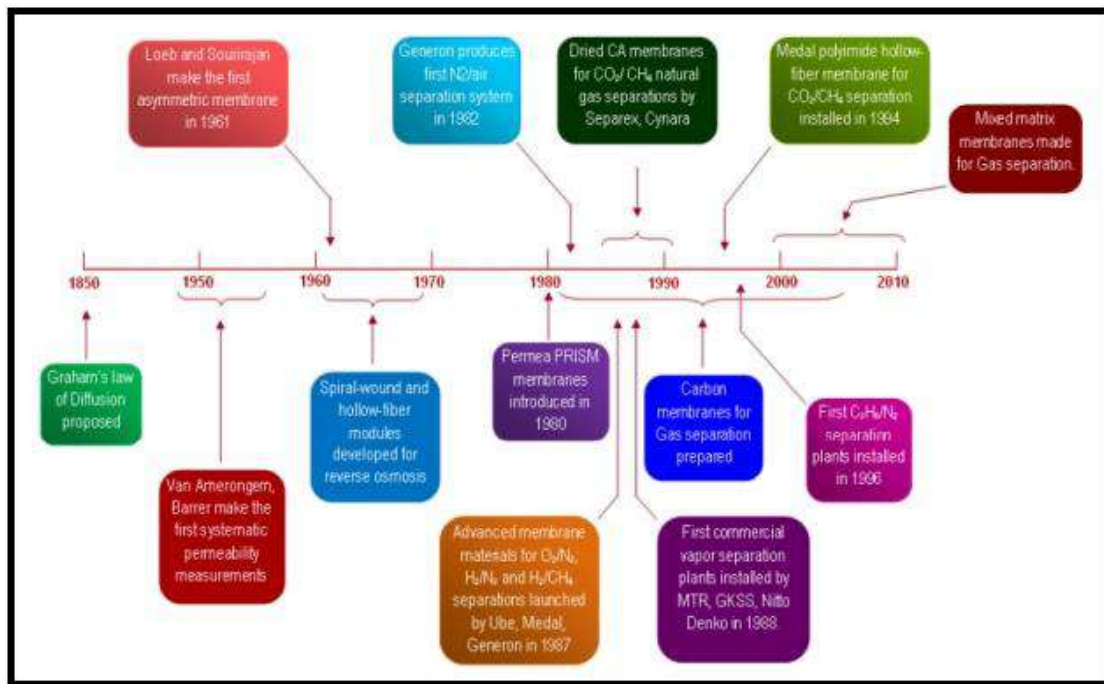
## **2 CHAPTER 2. LITERATURE REVIEW**

### **2.1 BACKGROUND HISTORY**

Early in the late 1960s, there was a surge in interest in membrane separation techniques due to two advances: first, the capacity to mass produce high flux, nearly flawless membranes; and second, the ability to mould these membranes into condensed, high-surface-area, cost-effective membrane modules. The technique was changed in the 1980s to accommodate different membrane processes [9].

Abbe Nollet's observation of the osmosis phenomena in the middle of the seventeenth century marks the beginning of membrane-based separations in written history. Fick developed his Law of Diffusion through Membranes more than a century later. Films can be utilized to distinct one gas from other gases, liquids from other liquids, dissolved particles from liquids, and suspended solids from liquids. Membranes can be made from a variety of organic (polymeric) and inorganic (ceramic) materials [19].

The greatest and first industrial use of gas separating membranes was for the separation of hydrogen gas in the 1980s by the firm Permea. Although it was recognized that membranes could separate gases in the 1980s, the technology was not yet advanced enough to affordably manufacture high performance modules and membranes. For RO applications, hollow fiber and spiral wound modules with large surface area membranes experienced rapid development in the early 1970s. In the future, Permea improved this technology to create Polysulfone Hollow Fiber Membranes that are specifically used to separate nitrogen from flue gases. Other businesses, including GMS, Cynara, and Saparex, used cellulose acetate membranes to get rid of CO<sub>2</sub> from the natural gas in the middle of the 1980s. For the purpose of separating N<sub>2</sub> from air, Generon developed the first air separation membrane system [20].



**Figure 2-1** History and milestones in membrane gas separation technique [19]

## 2.2 VARIOUS APPROACHES REGARDING FLOW VIA MODULE

Using CFD approaches, the effects of various spacer designs on flow patterns are examined by Asim Saeed et al. They claimed that since spacers speed up mass transfer and reduce the concentration polarization effect, they have a significant impact on module efficiency. By positioning the feed spacer at various angles to the intake feed flow, they examine three situations. In each example, the ratios of filament mesh size to height channel and filament diameter to height channel were 3.6 and 0.5, correspondingly. The inclusion of a feed spacer worked to support backwashing by producing a secondary flow pattern or cross flow velocity. The feed spacer's orientation has a significant impact on the drop of pressure and shear stress. Thus, improving the feed spacer's direction might improve membrane performance [21].

Two membrane modules—one standard and the other with high density—were put through a field test by A. L. Lee et al. to separate  $\text{CO}_2$  from natural gas. They operated in East Texas from 1993 to 1994 for over 20 months using a single stage spiral wound membrane module. They also used ideal cross-flow and mixing circumstances for the separation of gases to assess the field data. As a result, using membrane processing, natural gases with high  $\text{CO}_2$  concentration (up to 25 mole percent) may be transformed into pipeline-quality natural gas. The loss of methane in this study might be minimized to



roughly 10% using a single-stage system by employing fewer membrane sections. By counting a 2<sup>nd</sup> stage in sequence, the drops may have been condensed even further [22].

Salman Qadir et al. used a computational fluid dynamics (CFD) technique to consider the flux rate and gas transportation by means of membranes, as well as the instabilities in gas flow in modules. CFD approaches were used to explore module constraints such as length, flow rate of feed, permeate, pressure of feed, thickness of the membrane, and feed spacers. They claimed that greater permeation and higher flux were seen as the feed pressure rose. As a consequence, the permeating gaseous product has a high purity. Due to increased penetration purity, they believed that concentration polarization had minimal effects. By linking the CFD results with numbers from the literature, Arshad Hussain et al. were able to confirm their findings [23].

The feasible spacers in spiral wrapped membrane modules were upgraded by F. Li et al. in 2002. Commercial net spacers' mass transfer coefficients and power needs have been determined using CFD simulations. In the simulations, stable flow behavior, longitudinal and transverse vortices, and vortex shedding are seen, which favor mass transmission in the channels filled with spacers over vacant channels. Thus, CFD simulation results showed that the stream among two neighboring strands contains mostly of one "dead eddy," that does not significantly subsidize to mass transfer, when  $l/h$  is small (i.e.,  $l/h = 2$ ). The flow amongst adjacent strands is categorized by the shedding of vortices at an uncertain rate of  $l/h$ , or  $l/h = 4$ , which will significantly improve mass transmission. If the rate of  $l/h$  is relatively increased, such as  $l/h = 10$ , less average mass transfer will occur. Vortex shedding in this situation will merely enhance mass transfer near the filaments. This demonstrates that there is an ideal  $l/h$  ratio [24].

### **2.3 PERFORMANCE ANALYSIS OF SPIRAL WOUND MODULE**

The operating conditions, design, and preparation of spiral wound membrane modules and spacers were among the numerous parameters that J. Schwinge et al. observed in 2003. His research concentrated with spiral wound module performance analysis and optimization techniques. By examining the pattern of spiral wrapped modules and arrays using a grouping of tentative and computational approaches, the optimal array topologies, module geometries, and spacer designs for particular things may be discovered. The critical flux, sometimes referred to as the critical point of beginning of fouling, is said to indicate the onset of fouling when it comes to reducing fouling inside

modules and arrays. As a result, the spacer properties, such as the mass transfer and pressure loss, must be taken into interpretation for SWM optimization in direction to attain a maximum mass transfer rate at the minimum pressure loss. Today's easy access to CFD programmers and improved computer power and capacity, which were formerly the preferred method, are assisting in the creation of an essential determination of flow, shear, and pressure loss in modules as well as the pattern of novel spacers [16].

In 2015, Torsten Brinkmann et al. make a substantial progress in the large commercial scale separation of CO<sub>2</sub> utilizing membranes. Investigated of the possibility of separating CO<sub>2</sub> from biogas and hydrocarbon streams was done using a thin membrane composed of the block polymer PolyActive™ (methane). Modules containing the PolyActive™ composite membranes were employed for pilot-scale research. They found that PolyActive™ composite membranes exhibit robust carbon dioxide permeance at high CO<sub>2</sub> /N<sub>2</sub> selectivity and moderate selectivity of carbon dioxide towards hydrocarbon gases. They also showed that membrane surfaces up to 1 m<sup>2</sup> may fit within the 100 mm envelops of the spiral-wrapped module. These envelop modules produced 950 m<sup>2</sup>/m<sup>3</sup> packing density, however it varied depending on the feed spacer and envelop thickness [25].

Witopo Salim et al. constructed spiral wound membrane modules in 2018 and verified them in the field to recover CO<sub>2</sub> from flue gas utilizing "O" rings as face compression. As real flue gas was used in the field test at the National Carbon Capture Centre in Wilsonville, Alabama, the study of the modules discovered several unpredicted problems, such as glue line leaks and feed spacer dents on the membrane selective upper layer. The spiral-wound membrane module was produced with a smooth and soft feed spacer and an extended glue curing time in order to address these problems. The modules' strong probable for post-combustion CO<sub>2</sub> collection from coal-fired power stations has been revealed through successful field testing of the devices [17].

Kae K. Chen et al. enhanced the transportation membranes that segregate CO<sub>2</sub> from flue gas and are subsequently incorporated into SW modules in 2019. To increase the membranes' surface area, they developed a multi-membrane rolling approach with seven membrane leaves on each prototype module. It was possible to produce a full-sized module with a high packing density that ranged from 8" to 40". The packing compactness of the prototype modules was amplified by monitoring the thickness of the feed spacer.

These constructed SW modules allowed for the achievement of  $\text{CO}_2 / \text{N}_2$  selectivity of 185 and  $\text{CO}_2$  permeability of 1450 GPU while maintaining a pressure drop of 1.5 psi/m on the feed side permeate side. Concentration polarization and pressure drops were the most prevalent occurrences in SW modules that were controlled by quantitatively correlating with the input flow flux [26].

For separation on a large scale, membranes are usually fabricated in either hollow wound module or spiral wound module. Ana Carolina S. Dias et al. presented a scientific model to narrate the process of gas separation through spiral wound module. They studied common separation such as ammonia,  $\text{H}_2$ ,  $\text{CO}_2$  from natural gases as well as from flue gases through their proposed two-dimensional model. They also reported a numerical procedure for solving the models. The presented model accurately describe operation in SW module and the outcomes were very similar to literature up to some extent and the accuracy in results demonstrates the strength of 2-D model. It also provides an ease to predict concentration polarization and flux through SW module. The presented solving strategy was suitable than the other method because of their efficiency and calculations done faster [27].

The goal of Abdul Aiman Abdul Latif et al. work is to create a SWM separation model for mixtures of multielement natural gas. Within the multicomponent SWM module, the separation process is discretized using the succession stage approach in order to assess the pureness of product, loss of hydrocarbon, stage cut, and permeate acid gas conformation. Our findings imply that, in comparison to binary systems, multicomponent systems typically produce products with improved pureness of product, lower loss of hydrocarbon, and enhanced permeate acid gas conformation. Additionally, reliant on the element of the acid gas, various multicomponent systems produce varying parting performances. The spiral wound membrane system can be designed and optimized for industrial use using the established multielement SWM separation model. This is as a result of the binary combination having a higher methane component [28].

The most frequent undesirable impurity in natural gas is carbon dioxide ( $\text{CO}_2$ ) gas and methane ( $\text{CH}_4$ ) gas are the main product. Its presence alters the energy and heat content and values, leading to serious repercussions such pipeline damage and sharp increases in the cost of gas transportation. In order to use biogas as an alternative for natural gas,  $\text{CO}_2/\text{CH}_4$  break is also essential. Hence, the decrease in  $\text{CO}_2$  is seen as a significant

development in the sectors and has been the subject of various research. There are several techniques for separating CO<sub>2</sub>, including membrane separations, cold distillation, adsorption, and absorption. The membrane separation method for CO<sub>2</sub>/CH<sub>4</sub> separation is now the subject of substantial research [29].

Different polymers and different solvents are for the manufacturing of polymeric membranes, (polymers: polyethylene glycol, PDMS, and polyurethanes. Solvents: chloroform, tetrahydrofuran, N-methyl-2-pyrrolidone, methanol, and hexane etc). Roba M.A et al. Primary goal is to create PSF membranes by using tetrahydrofuran and chloroform as casting solvents. The synthesized membranes will be characterized using a variety of methods under the various circumstances. The study will also examine how the injection pressure and PSF structure made of vaporized solvents impact the penetration and selectivity of both CO<sub>2</sub>/CH<sub>4</sub> gases [29].

This research studies the outcome of two diverse solvents, on the production of PSF membranes. The outcome demonstrated that these membranes significantly affect how the CO<sub>2</sub>/CH<sub>4</sub> mixture separates. By utilizing the Robeson upper bound approach, the findings also showed that employing Casting solvent THF as presented greater efficiency in partition operation for both CO<sub>2</sub> and CH<sub>4</sub> comparable to CF membranes. According to the findings, increasing feed pressure had a negative impact on Permeability and membrane selectivity made with various solvents. Also, it was determined that the values of permeation for CO<sub>2</sub> is 62.32 and for CH<sub>4</sub> is 2.06 bar roughly at 1 and 2 bars when the membrane was created using THF. The permeability values are 57.59, 2.12 for CO<sub>2</sub>, CH<sub>4</sub> is 2.12 bar in CF, correspondingly. Furthermore, values of THF and CF's selectivity were 48 and 36 for both membranes at 1 bar, respectively. In light of this, it may be said that a membrane cast with THF performs better than one cast with CF. so, It follows that both are effective employed in the fabrication of PSF membrane have a considerable influence on the ability to segregate CO<sub>2</sub>/CH<sub>4</sub> gases from mixtures and have the possibility of being utilized on a wide scale [29].

In general, acceptance, the adsorption, cold extraction, and separation along membranes can reduce CO<sub>2</sub> gas discharges. Mono-ethanolamine can be used to separate CO<sub>2</sub> through the absorption technique. The absorption technique for separating CO<sub>2</sub> gas offers benefits over other methods since it uses less energy, has a large capacity for absorption, and is versatile. Nevertheless, using this process is expensive and calls for a specific solvent.

Either chemical or physical adsorbents can be used in adsorption techniques for CO<sub>2</sub> gas separation. The adsorbents Zeolite (CaX/Ceca 13X) have been studied for CO<sub>2</sub> separation [30].

Songolzadeh et al. noted that despite the fact that adsorption process consumes less energy, it has a limited adsorption capacity, is difficult to regenerate adsorbents, and needs more study to identify new adsorbents. Li et al. have studied the cryogenic distillation method for gas separation, which uses technology that is both industrially applicable and reasonably simple to build. Moreover, this process doesn't generate liquid CO<sub>2</sub> or employ solvents. Yet it takes a lot of energy to chill and condense the Carbon dioxide at low temperatures, which can lead to additional operational issues. Gas pressure affects gas extraction using membrane technology. Due to factors including the use of straightforward instruments, unambiguous procedures, excellent permeable and specificity, excellent chemical and thermal durability, resilience to plasticization, and cheap production costs, this method offers benefits above other CO<sub>2</sub> gas separation systems.

Due to these shortcomings, Witri W.L et al. focused on creating alternative methods for creating membranes which are more reliable, affordable, and have a high level of extraction efficiency, namely through the use of membrane materials combinations called mixed matrix membranes. Compared to polymeric membranes, the mechanical properties produced by MMMs' manufacturing method are expected to be more stable and to have greater separation capabilities. The continuous phase of MMMs contains a polymerized organic matrix, while the dispersion layer contains filler particles. Polyethersulfone is one polymer matrix with promise for making MMMs (PES). Because to its strong mechanical characteristics, superior chemical and thermal durability, and significant T<sub>g</sub> near 225°C, this polymer is frequently employed in the production of membranes. MMMs that based on PES achieved the best efficiency for separation of carbon dioxide/methane 41.42 and CO<sub>2</sub> permeability at 10.11 with the addition of ethylenediamine (EDA)-TiO<sub>2</sub> 5% at pressure (4 bars). Moreover, PES/Zeolitic Imidazolate Framework-8 (ZIF-8) that is MMMs used for the separation of H<sub>2</sub>/CO<sub>2</sub> and H<sub>2</sub>/N<sub>2</sub> gases, with selectivity values of 9.3 and 11.5 respectively. With inclusion of fifteen percent carbon molecular filter in PES membranes, carbon dioxide/methane gas selectivity changes 3.57-11.15. MIL-100, which also uses aluminum (Al). Volkringer et al. As MIL-100(Al) filler has never been utilized in separation applications, this study studied how MMMs might be constructed using

MIL-100(Al) and PES polymers in the hopes that this would increase the MMMs' capacity for CO<sub>2</sub> gas parting. Witri W.L et al. Furthermore, as compared to pristine PES membranes, The penetration of CO<sub>2</sub>, O<sub>2</sub>, and N<sub>2</sub> gases may be increased by 16, 26, and 14 times, correspondingly, by adding MIL-100(Al) to PES. Additionally, the specificity for Carbon dioxide and oxygen gas separation may be raised from 2.67 to 4.49 by adding 20% MIL-100. When we use of 10% MIL-100 as filler, carbon dioxide/nitrogen gas selectivity may be raised 1.01-2.12.

There is also a discussion of multiple polymers under studied for gas segregation, mostly to address issues now present or gain access to applications not yet used commercially. Families of polymers that was been the attention of a lot of recent study comprise the thermally rearranged (TR) polymers, PIMs (polymers of intrinsic microporosity). RTILs (room-temperature ionic liquids), per fluoropolymers, and raised polyimides [31].

The significance of polymer research in current and future gas materials extraction processes is the main concern of David F.S et al. In order to lay the groundwork for the discussion of advanced materials, current applications of polymers in gas separation methods are examined along with potential future applications that may be enabled by improved membrane materials [31].

The first technological advance for separation of carbon dioxide/hydrogen sulphide was comprehended by cellulose acetate base membranes, which, at that time, were mostly employed in an uneven form for desalination. Early natural gas deacidification experiments utilizing cellulose membranes date back to the late 1960s. The spiral wrapped modular design of dry cellulose acetate membranes showed a mixed gas selectivity that was much lower selectivity that predicted from unmixed gas tests, showing plasticization impact due to carbon dioxide and other hydrocarbons. Mazur and Chan show that CA membranes can remove 95%/90% of CO<sub>2</sub>/H<sub>2</sub>S, individually. Schell and Houston found that CO<sub>2</sub> had a selectivity of 20-30 over CH<sub>4</sub>, while H<sub>2</sub>S had a selectivity of 75-110 over propane [32].

For acid gases other polymers have better performance in term of selectivity when it is using as membrane material, such as polysulfones, polyimides and polycarbonates, have drawn interest from S. Sridhar et al. in the 1980s.

Robeson conducted systematic research on variety of glassy and flexible polymers to determine impact of additives on gas diffusivity. The existence of flexible siloxane linkages and their capacity to accept a variety of additives onto the chain of polymer, it has been revealed that silicone polymers are attractive materials for investigating gas separation properties [32].

Because silicone rubber has stronger gas penetration capabilities than other synthetic polymers, it was once thought to be a prime contender for gas separation. This was the case up until 1983. The enormous free volume of silicones has been linked to their high permeability; this may be because the siloxane links within the polymer are flexible.

It was decided to go from rubbery to glassy polymers while also making an effort to boost the latter's permeability. The permeability of aryl polymers, such as polysulfone, was improved to some extent by adding  $\text{CH}_3$ . Tetramethyl bisphenol A polysulfone was investigated by Stannett et al. They found that it preserved selectivity while increasing gas permeability. Due to poor residual volume, lack of continuity, less bulk weight, and low  $T_g$ , polysulfones generated comparatively worse selectivity despite their best efforts. The ability of polyether sulfones (PES) to pack more densely than related polysulfones in the glassy state has prompted researchers to look into their straight and short chain architectures. PES membranes had somewhat greater selectivity than polysulfones, with carbon dioxide and methane for PES being 28 as opposed to 26 [32].

Sandru et al. claim that plate-and-frame type of module having 1.5 m<sup>2</sup> area was made using polymeric polyvinylamine (PVAm) assisted transport membranes. It was shown to be both practical and long-lasting enough to be used continuously, genuine flue gas, but at the highest  $\text{CO}_2$  load of 75% in the permeate,  $\text{CO}_2$  extraction was only 0.09% - 0.36%. In order to conduct research in a pilot plant, Pohlmann and teammate fabricate a membrane module by using 12.5 m<sup>2</sup> of multilayer membranes of material PolyActive TM. It was reported that this membrane module using PolyActive TM membranes can recover 42.7 percent carbon dioxide and can produce 68.3 mol% pure  $\text{CO}_2$ , which is tremendously hard to perform on a large scale, at an optimal permeate level as low as 0.05 bar [15].

Yoo et al. looked at a unit using plate-and-frame type of module with an operative membrane area of about 1080 cm<sup>2</sup>, which could achieve at a feed flow rate of 0.2 Nm<sup>3</sup>/h a 66 mol% pure  $\text{CO}_2$  and a  $\text{CO}_2$  extraction rate of 38% and a fixed permeate value of 0.2 bar. A complete manufacturing process for hollow fiber membrane modules with high

packing density was proposed by Li et al. The pilot membrane system described by He et al. used two parallel hollow fiber membrane modules with 4.2 m<sup>2</sup> of membrane area each. The CO<sub>2</sub> produced by their membrane method was 54%–56% pure and had CO<sub>2</sub> recovery rate of 0.9%.

In addition, Deng and colleagues fabricate a 200 cm<sup>2</sup> hollow fiber membrane module for lab-scale testing in a cement mill. One tonne of CO<sub>2</sub> is said to be able to be processed per day by a membrane separation unit utilizing spiral-wound type of modules that is also disclosed. Ho and colleagues also succeeded in fabricating and testing of SW modules for assisted transport having roughly 300 cm<sup>2</sup> to 2.94 m<sup>2</sup> total membrane areas on a lab-scale. In industrial applications there is an urgent need of installing the module that should have high performance, which should optimized manufacturing procedures and operational plans. Additionally, it is essential to research membrane modules to sustain the gap between system engineering and membrane development.

In comparison to the modules such as hollow-fiber and plate-and-frame types of module, SW membrane modules, which are based on easily obtainable flat sheet membranes, have the potential to be the most widely used method of post-combustion CO<sub>2</sub> collecting. Previously, a pressurized two-stage membrane pilot plant's ability to extract CO<sub>2</sub> was thoroughly examined using three simulated exhaust gases. Actual plan of a three-stage, three-step membrane separation unit for extracting CO<sub>2</sub> from coal-fired power plants exhaust fumes was then demonstrated. The large-scale SW membrane module hasn't drawn much attention to its preparation process or performance.

Additionally, the operating strategy for the membrane module should be optimized for the driving force mode and connecting mode (such as series or parallel). The spiral-wound membrane module, which had up to 31 m<sup>2</sup> an operative membrane, was the subject of this study, which aimed to demonstrate a methodical development procedure for it. The effects of pressures at which system operates and the feed conditions would then be looked at. Based on the outcomes of module arranged in various mode situations, a multi-core membrane module was created and constructed that hold up to 14 membrane components. This work is expected to help with the practical application of membranes in post-combustion capture [15].

Common solvents can dissolve PU and PES, indicating that the co-casting approach has a reasonable chance of producing dual-layer membranes. Tetrahydrofuran (THF) and N-



methyl-2-pyrrolidone in a solution were used by Pereira et al. to create dual-layer membranes, with NMP serving as the solvent for the discerning dope and THF serving as the solvent for the porous support dope. The permeable and selective layers were both constructed using PEI and PES. They looked at the casted solutions' exposure period (before to immersion), as well as its impact on membrane architecture. The membranes, however, did not exhibit strong adherence or homogeneity. NMP was used as the solvent for both doped solutions by Mei-Li et al. to study breakdown in polyetherimide/polysulfone hybrid-layer membranes and to increase the adhesion between the layers. They discovered that both layers should contract proportionately with closer percentage values following coagulation in order to increase adhesion between them. By using the co-casting technique, dual-layer PU/PES membranes which intended to remove Carbon dioxide from the flue gases were effectively created.

Dual-layer membranes might be created by combining two dissimilar materials using comparable solvent compatibility. Impacts of the mixing tub temperature and solvent evaporation time were established, and they demonstrated that evaporating for 15 seconds and a water combining bath at temperature 75 °C, as shown by Scanning electron microscopy (SEM), a good multilayer adherence and a homogeneous support structure were created. For the both membranes i.e. dense PU and dual-layer PU/PES membranes, the normalized effective permeability of the individual components of flue gases such as, nitrogen, oxygen and carbon dioxide was computed [33].

Although the rates of permeability for dual-layer membranes were lesser, the permeability rates for the single-layer membranes were greater because the support which was porous offers an extra mass barrier to the membrane. Permeability of all gases decreased in permeation experiments at feed pressures between 1 and 8 bars. Since the incorporation of CO<sub>2</sub> into PU was further compromised in that pressure range, the CO<sub>2</sub>/N<sub>2</sub> optimum selectivity dropped from 66 to 60. On the contrary hand, experiments carried out between 25 and 45 degrees Celsius revealed an improvement in gas permeability with temperature because more free volume was accessible in the dense layer. However, the selectivity of CO<sub>2</sub> to N<sub>2</sub> dropped from 66 to 43 in the temperature range of 25-45 degree celcius because the impact is added significantly for gases with greater kinetic diameters (nitrogen). The manufacture of selected dual-layer membranes for gas separation could benefit from the optimization of the co-casting technology, which will enable the utilization of adaptable combinations of materials for the layers [33].

### 3 CHAPTER 3. EXPERIMENTAL WORK

#### 3.1 MATERIALS USED FOR MODULE PREPARATION

For synthesis of membrane solution beakers, stirrer, hot plate, weight measuring machine, sonicator, and, drying oven. For casting of membrane micro adjustable Film applicator has been used.

**Table 3-1** Material used for making membrane solution

Sr.	Material	Specifications	Company	Chemical structure
1	Polyether sulfone	<ul style="list-style-type: none"> <li>Grade= ULTRASON® E6020P</li> <li>apparent density = 0.25g/cm<sup>3</sup></li> <li>Physical form = flakes</li> <li>Molecular weight = 72000g/mol</li> </ul>	Ultrason®	
2	N, N-Dimethylformamide	<ul style="list-style-type: none"> <li>Density = 0.94 g/cm<sup>3</sup></li> <li>Color = colorless</li> <li>B.P = 152 degree C</li> <li>Form = liquid</li> <li>Grade = reagent grade</li> <li>Storage temperature = ambient</li> </ul>	Honeywell	

For casting of membranes:

**Table 3-2** Material used for casting membrane

<b>Sr</b>	<b>Material</b>	<b>Specifications</b>	<b>Source</b>
1	Baking sheet (non-woven PP)	<ul style="list-style-type: none"> <li>It should not be chemically reacted with solution i.e., inert</li> <li>Should be Heat resistance</li> </ul>	Freudenberg Germany
2	Paper tape	<ul style="list-style-type: none"> <li>It should have strong adhering power</li> </ul>	Department
3	Measuring tape		Department
4	Scissor		Department

For the fabrication of module:

**Table 3-3** Material used for fabrication of module

<b>Sr.</b>	<b>Material</b>	<b>Specifications</b>	<b>Source</b>
1	Perforated plastic tube	Length = 12" Material = Polypropylene Outer diameter = 16.94mm Inner diameter = 10.05mm Number of perforations = 10	DADA Enterprises (Pvt) Limited
2	Carrier sheet	Teflon Tape	Local market
3	Epoxy resins and epoxy hardener	It should be flexible after curing It should be moisture resistance It should be inert, no reaction with gases	Belite's no 1
4	Polyethersulfone membranes		Synthesize in laboratory
5	Feed spacer	Material = Polypropylene	DADA Enterprises

			(Pvt) Limited
6	Permeate spacers	Material = Polyester	DADA Enterprises (Pvt) Limited
7	Gaskets	Material = Rubber	
8	Rubber O-rings	Material = Rubber Outer diameter = 18.2mm	

## 3.2 METHODOLOGY

### 3.2.1 PREPARATION OF MEMBRANES

#### Solution

Take 20 grams of polyether sulfone (PES) and 80 ml of N, N-Dimethylformamide (DMF), which is 20% weight/volume of total solution. Pour DMF solvent and a stirrer in a beaker than put the beaker on hot plate at almost 100 °C. Add PES into the beaker little by little and stirrer it for at least 24 hours to make a perfect and dissolved solution. After that, sonication of solution is done for an hour to remove air bubbles and any gases entrapped in the solution.

Sonication is a device that is used to generate high frequency sound waves that used for various purposes. It is fully automated system having temperature and time regulated. It consists of stainless-steel lid in order to lessen the frequency of sound waves in atmosphere. There is a thermostat which regulate the temperature of Sonicator. There is an ultrasonic button that is switch-on to produce high frequency ultrasonic waves. It is used to disperse the coagulated solute in solvent as well as to remove the air bubbles entrapped in solutions.

#### Casting of membrane

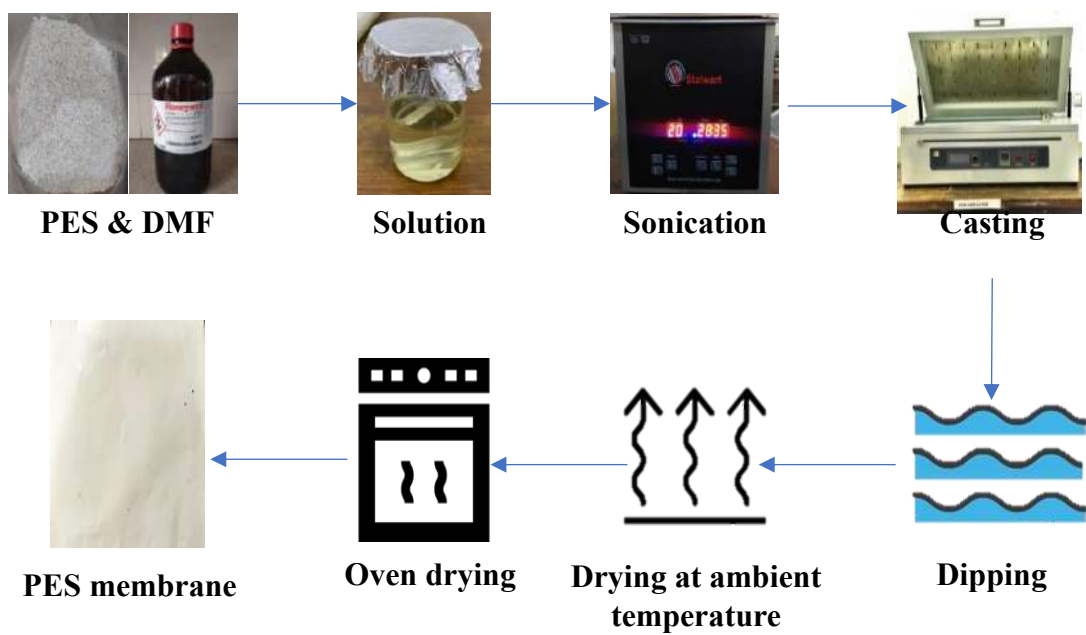
The film applicator is designed to produce a film of controlled thickness. There are different types of film applicators available in the market that can be operated manually as well as automatically. When films are produced manually there is an excellent chance of human errors. Human errors cause inconsistency in the film. This

inconsistency based on various aspects counting the speed above which the film applicator is moving, how smoothly you draw down the film applicator, and also the downward force you are applying on the film applicator during casting. In this type of film-casting machine, two micrometers attach to the doctor blade through which the gap is adjusted to make a film of desired thickness. In this casting machine, there is also a heating system that allows the complete elimination of solvent and temperature can be adjusted according to need. There is a glass plate on which the film is cast. These applicators are used to make dense membranes and are perfect for fluids having high viscosity. Now we will talk about the methodology.



**Figure 3-3** Doctor blade in Department of Polymer and Process Engineering (Polymer Composite Lab), UET Lhr

Take a non-woven PP baking sheet of 14” width and 24” length and attach it to the glass plate in such a way that no air entrapped in between the baking sheet and glass plate. Set thickness of 120 microns on doctor blade and set on the glass plate properly. Pour the solution on glass plate in front of film casting doctor blade and cast a film by moving doctor blade slowly across the substrate. Then after 30 seconds, immediately dip the membrane in cold water. Dip it for two minutes and then take out of water. Let the membrane to dry for one day at ambient temperature. After 24 hours, put the membrane in the drying oven at 60 degrees Celsius for complete drying. After every hour, elevate 30-degree Celsius temperature of oven up to 140 for complete removal of solvent from the membrane. Now, membrane is ready for fabricating into the spiral wound module.



**Figure 3-4** Schematic of the methodology of membrane preparation

### 3.2.2 SPIRAL WOUND MODULE FABRICATION

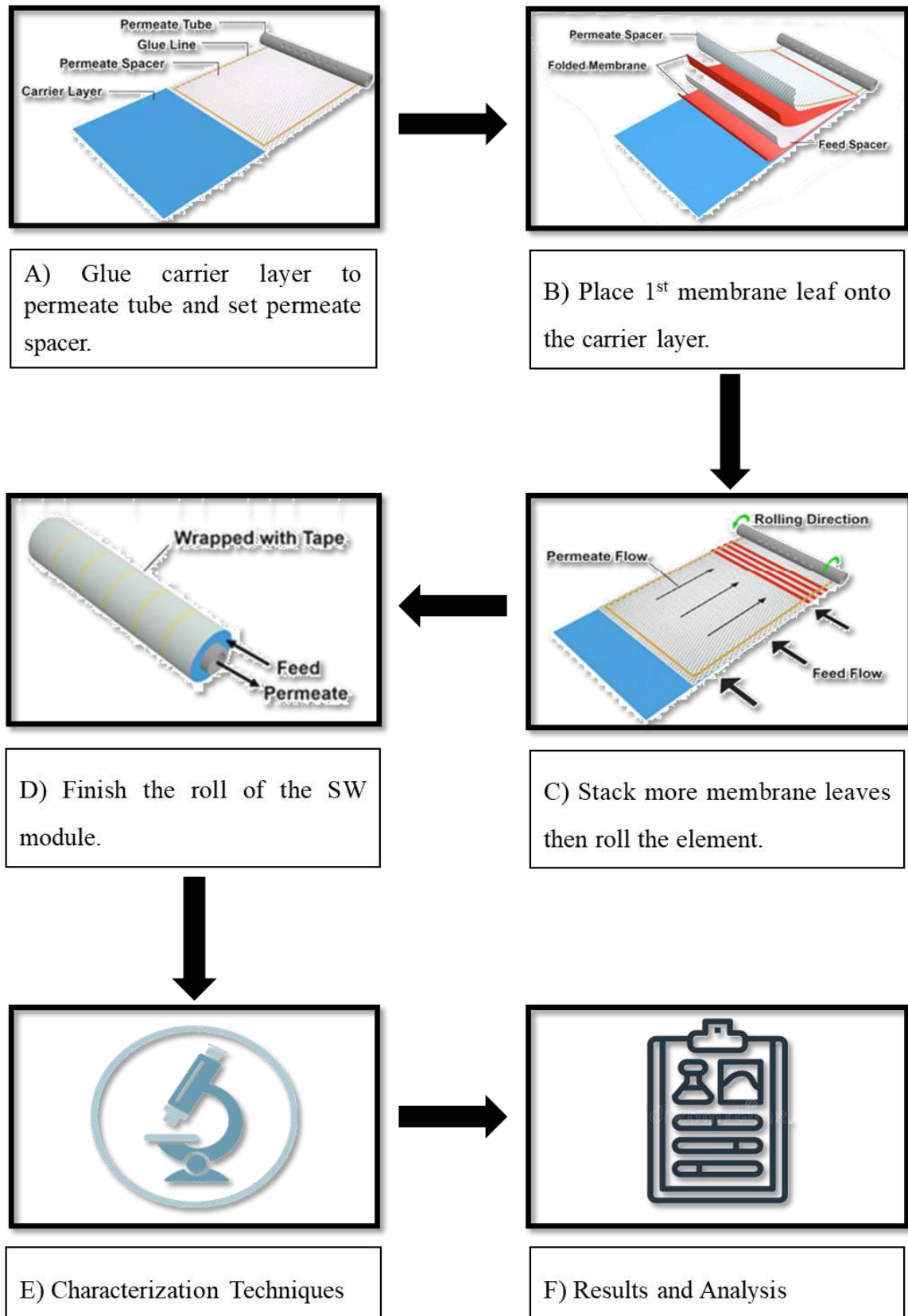


Figure 3-5 Methodology of fabrication of spiral wound module

### **3.2.3 CONFIGURATIONS OF SWM**

#### **1<sup>st</sup> CONFIGURATION**

Took a perforated tube and glued the permeate spacer of length 24" and width 10" around it but made sure that glue could not go in holes of tube. Then, one membrane was put beneath permeate spacer in the direction of upside down and other one was on upper side of permeate spacer by facing membrane side towards you. All membranes had length of 20" and width of 10". Glued all sides except the side towards tube, of both membranes properly having permeate spacer between them. Then, feed spacer of length 20" and width 10" was kept above this leaf without glue. Similarly, another leaf was made just as described above and it was put above that feed spacer. Then, again another feed spacer was put over it. Made sure that all the elements kept properly on one another. Without waiting of proper curing of glue, start wounding all the elements around perforated tube. Module should be tightly wound. Lastly, a sticky carrier sheet was put on the outside of wound to assemble all the elements together.

#### **2<sup>nd</sup> CONFIGURATION**

Made a leaf and glued it till center of membrane. Perforated tube was kept at that center. The other half side which was not glued, was then glued with other half side of leaf. Then, again one more leaf was taken and glued with other side of leaf and then glued it with the 1st half side of membrane leaf. Made sure that all the permeate spacers were properly glued with perforated tube, mainly on the upper edge and lower edge of tube. So that, there will be no leakage. Then, feed spacers were put between all the leaves. Three feed spacers were used in that configuration. Then, press all the leaves on one side and started wounding around perforated tube. Lastly, a sticky carrier sheet was put on the outside of wound to assemble all the elements together. In this configuration, all the dimensions of membrane, permeate spacer and feed spacer was taken as in 1st configuration.



### 3.3 CHARACTERIZATION TECHNIQUES

#### 3.3.1 GAS PERMEATION SETUP

The technique of gas permeation uses nonporous polymer membranes with selective gas permeability based on a dissolution-diffusion mechanism to fractionate gas mixtures. This permeation setup is used to conduct a permeation test using gases (CO<sub>2</sub>, H<sub>2</sub>, methane, and nitrogen) [34]. The pressure difference transversely the membrane drives the membrane gas separation process. In our experimental effort, we separate (CO<sub>2</sub>) gas to improve the safety and health of our environment and lessen the impact of the greenhouse effect.

We present a straightforward experimental technique for describing the H<sub>2</sub>, He, N<sub>2</sub>, and Ar diffused in polymers' gas permeation abilities. This depends on a more advanced diffusion analysis program and the volumetric measurement of liberated gas during high-pressure exposure. The passage of a permeate through the material membrane of a solid, such as a liquid, gas, or vapors, is referred to as permeation. Adsorption of the penetrating species into the polymer, dispersion through the polymer membrane, and desorption of the penetrating species from the polymer surface are the three processes that make up permeation. Permeation is essential for many design applications, such as gas separation, packaging, etc. [35].

To measure gas permeation, utilize the gas permeation test cell. The temperature and pressure of this sort of cell are typically maintained at atmospheric pressure. The feed side and the permeate side of the cell are divided by the test film. The gas which we want to capture, such as CO<sub>2</sub>, is continually delivered into the feed side. The other gas, such as nitrogen, is used on the other side to remove the permeate gas as it absorbs across the test film. The partial pressure of the permeate gas is saved almost zero by sweeping the permeate out of the cell. The quantity of permeate that has gone over the test film is calculated by analyzing the other gas and permeate gas stream. Based on the gas, different analytical techniques are used. There are various techniques to analyze for permeants[36]. Permeability can be determined by the equation given below [37].

$$P = \frac{22414}{RT} \times \frac{V}{A} \times \frac{l}{\Delta P_i} \frac{dP_i}{dt}$$

P = Permeability of film (m)     $\Delta P_i$  = absolute pressure difference of feed side and downstream in pascal

$V$  = volume of downstream ( $\text{cm}^3$ )     $A$  = Membrane active area ( $\text{cm}^2$ )

$\frac{dP_i}{dt}$  = slope of pressure vs time (pascal)     $l$  = Thickness (cm)

Selectivity can be determined by following formula;

$$\alpha = \frac{P_{CO_2}}{P_{N_2}}$$

Whereas,

$\alpha$  = Selectivity of membrane/module

$P_{CO_2}$  = Permeability of carbon dioxide gas

$P_{N_2}$  = Permeability of nitrogen gas



Figure 3-6 Gas Permeation Setup in Department of Polymer and Process Engineering (Polymer Synthesis Lab), UET Lhr

### 3.3.2 LEAKAGE TEST

In order to check any leakage in spiral wound module, we perform the leak test by using leak test apparatus. For this purpose, the membrane housing loaded with the spiral-wound module. Connect a dehydrated air tube that is associated with a rotameter to adjust the air stream rate to the housing's feed inlet. Before installing the spiral wound film module, turn off the valve to ensure there is no air movement. Join the pressure gauges to

the feed exit and permeate opening. Each pressure gauge's needle valve has to be left exposed to the atmosphere. Pad the permeate side's outer opening end. This makes sure that the gauge pressure attached to the permeate side's intake end can detect any rise in compression on the permeate side. As directed by the rotameter, begin the air stream to the feed intake at a degree of 1000 sccm. To raise the compression on the feed intake to 1–2 psig, steadily spin and turn off the needle regulator for the pressure gauge attached to the feed and retentate side. After that, totally seal the needle valve on the pressure gauge that is attached to the permeate side's intake end [26].

The gauge pressure on the permeate side will immediately detect any leak from the feed side to the permeate side because the permeate side's pressure will quickly rise and finally equal the feed side's pressure (1.5 psig in this case). This suggests that the spiral-wound module has a leak. If the permeate pressure gauge does not register a rise in pressure, examine all the connectors for any potential sources of leakage from the enclosure to the atmosphere. Use the soap bubble test on each connection as one method of checking for leaks in the connections [17].

### **3.3.3 FOURIER TRANSMISSION INFRARED SPECTROSCOPY (FTIR)**

FTIR (Fourier Transform Infrared Spectroscopy) is a powerful analytical performance that is widely utilized in various fields such as chemistry, biology, materials science, and pharmaceuticals. It was performed using JASCOTM FT/IR 4600 with 32 scans to study the composition of the pure polyether sulfone membrane. It predicts results in two regions which includes; fingerprint region and the functional group region, with absorption peaks that relate to the vibrational frequency between the bond of atoms. It is a non-destructive analytical method that provides information about the molecular composition of a sample by measuring its absorption or transmission of infrared radiation. Some of the significant applications and benefits of FTIR are; identification of unknown substances, quantitative analysis, structural analysis, quality control, and, environmental analysis [38, 39].



**Figure 3-7** Fourier Transmission infrared spectroscopy (FTIR) in Department of Polymer and Process Engineering (Polymer Characterization Lab), UET Lhr

### 3.3.4 THERMAL GRAVIMETRIC ANALYSIS (TGA)

Thermogravimetric Analysis, or TGA, is a potent analytical method for examining a material's thermal characteristics. It includes weighing a sample as it is heated or cooled under carefully regulated circumstances. To perform the procedure and evaluate the thermal stability of materials, a Shimadzu™ TGA-50 was utilized. The tests were carried out in accordance with ASTM-638 at a 10 °C/min heating rate. At a degree of 10 °C/min, the temperature was raised from 50 to 600 C. A TGA scheme was obtained using thermogravimetric data from a thermal reaction, with mass or % of original mass on the vertical axis and time or temperature on the horizontal axis. The plot's data can be used to calculate the purity, composition, drying, and ignition temperatures of a substance. Some of the significant applications and benefits of TGA are; determination of thermal stability, identification of composition, evaluation of purity, kinetic analysis, and, quality control [40].



**Figure 3-8** Thermogravimetric analysis Setup in Department of Polymer and Process Engineering (Polymer characterization Lab), UET Lhr

### 3.3.5 UNIVERSAL TESTING MACHINE (UTM)

Tensile tests, also known as tension tests, are carried out by tugging on a tensile specimen utilizing the UTM TIRA test 2810 E6 in order to determine how a material responds to tension forces. As the material is pulled, its tensile strength and degree of elongation are measured. The ASTM D412 was followed in the preparation of the samples. Numerous tests, including tensile, flexural, compression, peel, rip, friction, cycling, and modulus, can be handled by robust universal tensile testers [41].



**Figure 3-9** Universal testing machine Setup in Department of Polymer and Process Engineering (Polymer characterization Lab), UET Lhr

### 3.3.6 OPTICAL MICROSCOPY

In an optical microscope, an objective lens creates a magnified image of a specimen of an object, and an eyepiece enlarges the image even more so the user can view it without a lens. Direct light that has passed through the specimen undisturbed and light that has been diffracted by the specimen's small features interfere to generate an image at the intermediate picture plane [42].



**Figure 3-10** Samples utilized for optical microscopy

## 4 CHAPTER 4. RESULTS AND DISCUSSION

### 4.1 FOURIER TRANSMISSION INFRARED SPECTROSCOPY (FTIR)

The infrared spectrum of pure polyether sulfone in the range of 600-4000  $\text{cm}^{-1}$  is shown in figure 4-1 below, the vibrations that are seen at 1239.04, 1149.37  $\text{cm}^{-1}$  are sulfone groups, however the backbone vibrations at 1483.96, 1579.41  $\text{cm}^{-1}$  can be associated to benzene aromatic groups ( $\text{C}_6\text{H}_6$  group). The peak that are seen at 1011.48  $\text{cm}^{-1}$  is the peak of aromatic ether ( $-\text{C}-\text{O}-\text{C}-$ ) chemical bond. A weak vibration peak that is seen at 2920  $\text{cm}^{-1}$  is ( $-\text{N}-\text{CH}_3$ ) functional group that is also present and has been seen in DMF chemical structure. The peak at 3086.51  $\text{cm}^{-1}$  shows OH stretch [43, 44].

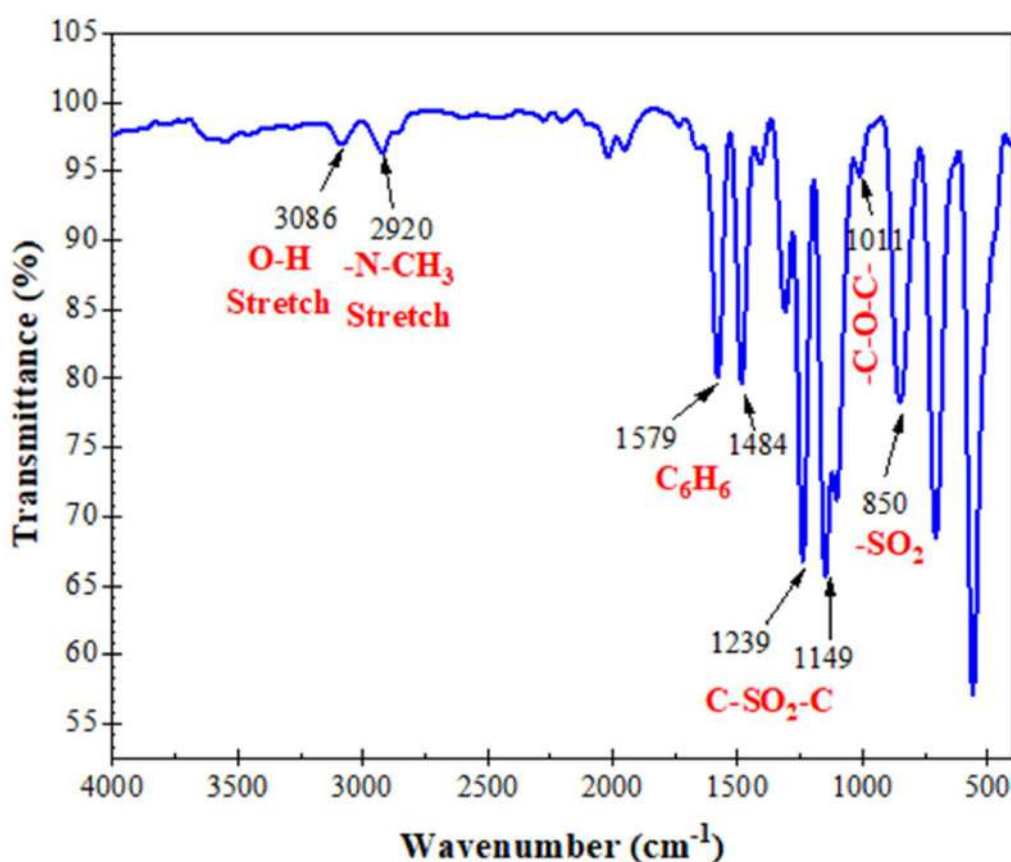


Figure 4-1 FTIR Analysis of PES membrane

### 4.2 THERMAL GRAVIMETRIC ANALYSIS (TGA)

TGA study was utilized to determine the thermal constancy of neat Polyethersulfone (PES). The analysis has shown that PES exhibit high thermal stability which supports with the earlier research by Witri et al. [30].

From graph 4-2, it has been demonstrated that mass reduction happened twice, once at temperatures 150°C and once at 519.68–552.93°C and these similar results have been recorded in literature. First drop at 150 degree C is not very steep because at this instance mass is reduced due the evaporation of DMF solvent present in very minor quantity (>2%) in PES membrane. While the second drop at 519.68 degree C is very steep. The major mass reduction occurs at onset temperature (519.68°C) which is degradation temperature of PES. Thermogravimetric analysis revealed that PES is thermally stable up to 519.68°C there is 49.926% weight loss from 150 to 593.21°C [45, 46].

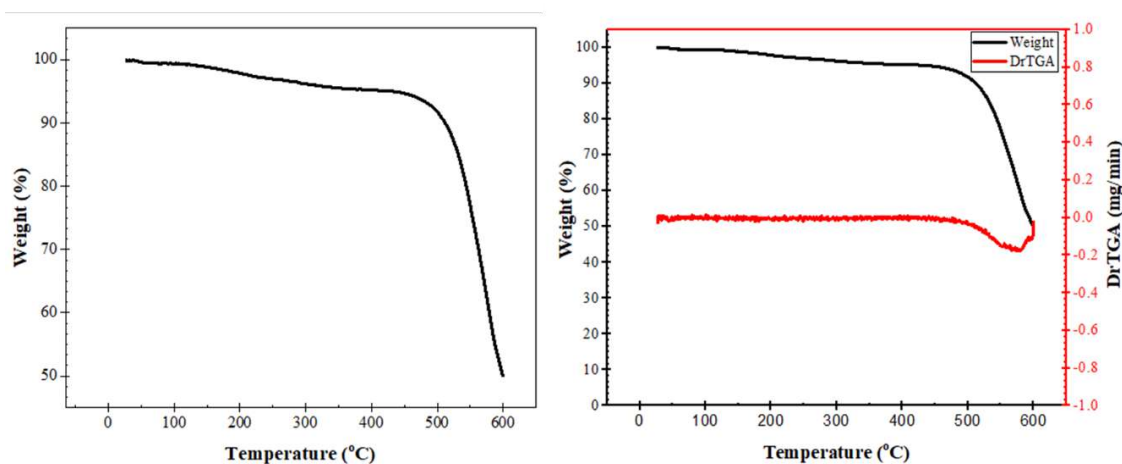
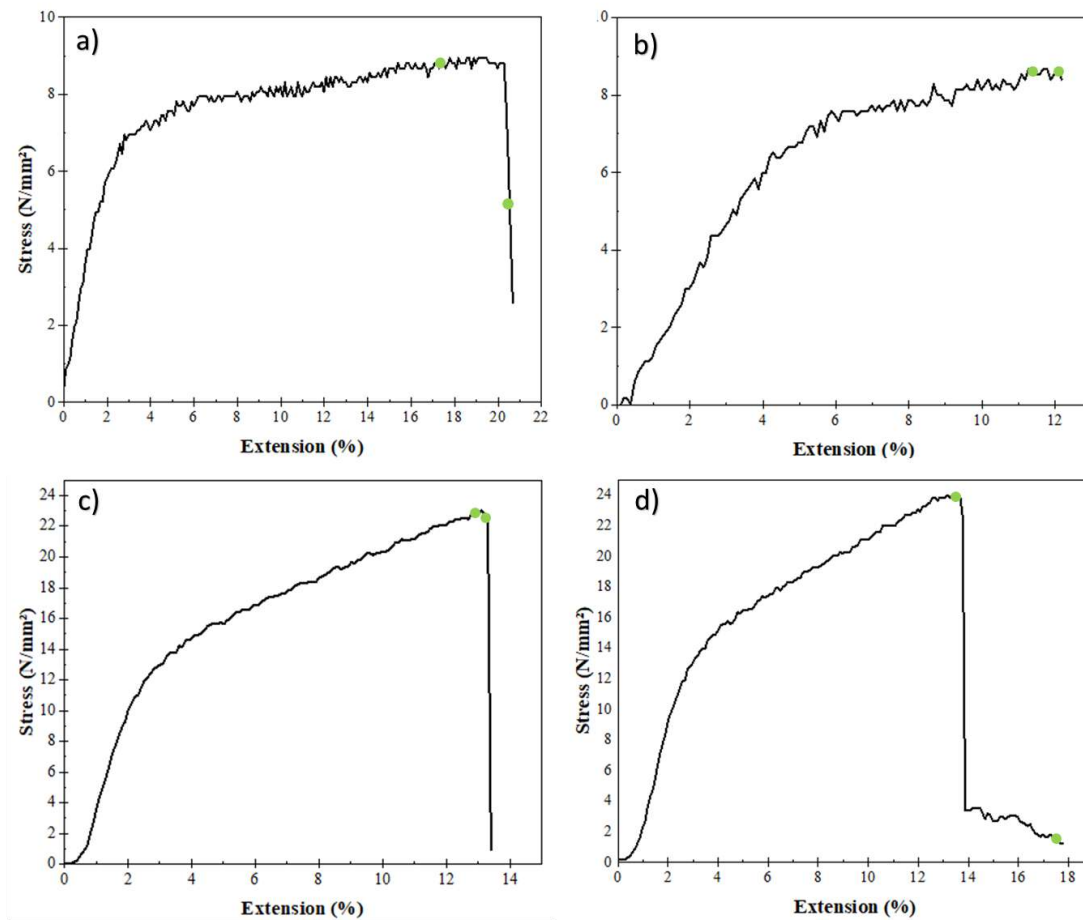


Figure 4-2 TGA Analysis for PES membrane

### 4.3 UNIVERSAL TESTING MACHINE (UTM)

UTM testing has been performed for checking the flexibility of membranes and the ability of membranes to bear stress. In graph **a**), the stress bear by a membrane is 8.94 N/mm<sup>2</sup> with an extension of 17.78% and in graph **b**), the stress bear by a membrane is 8.67 N/mm<sup>2</sup> with an extension of 11.28% whereas, in graph **c**), the stress bear by a membrane is 23.01 N/mm<sup>2</sup> with an extension of 12.91%. and in graph **d**), the stress bear by a membrane is 23.99 N/mm<sup>2</sup> with an extension of 13.17%. If a membrane bears large stress without large elongation, then, that membrane has good mechanical strength and strong chemical chains. Similarly, graph **c**) and **d**) having the support of substrate can bear large stress as compared to graph **a**) and **b**), and also have good flexibility property for wounding purpose. As in the spiral wound module, for bearing large pressure of the gas, the membrane with the support of substrate will be helpful [47].

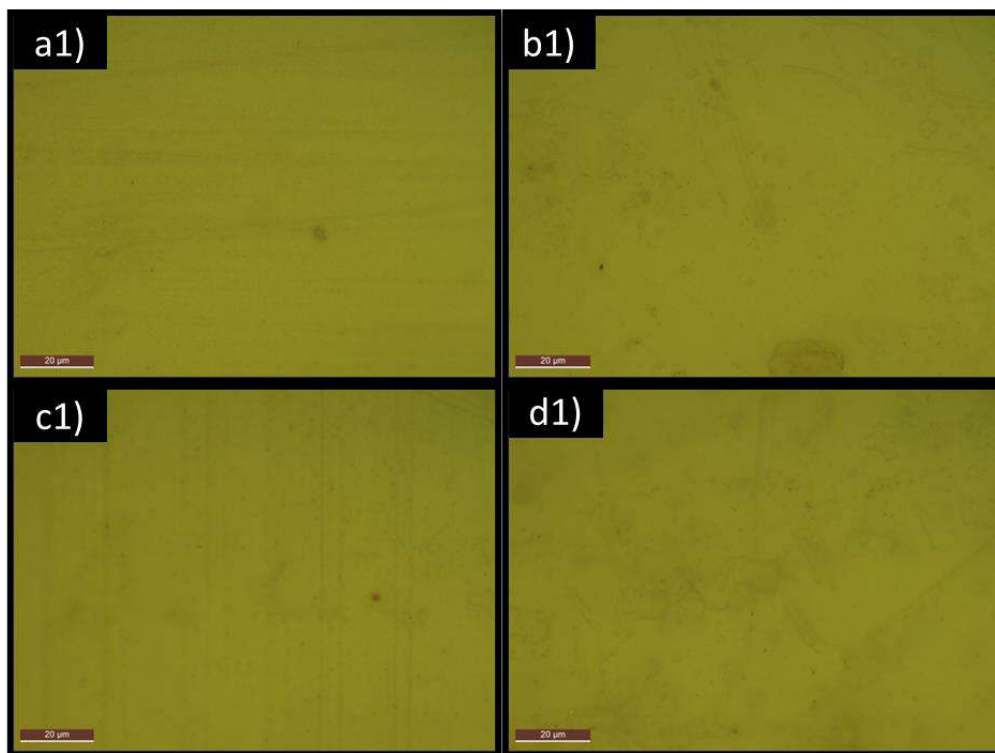




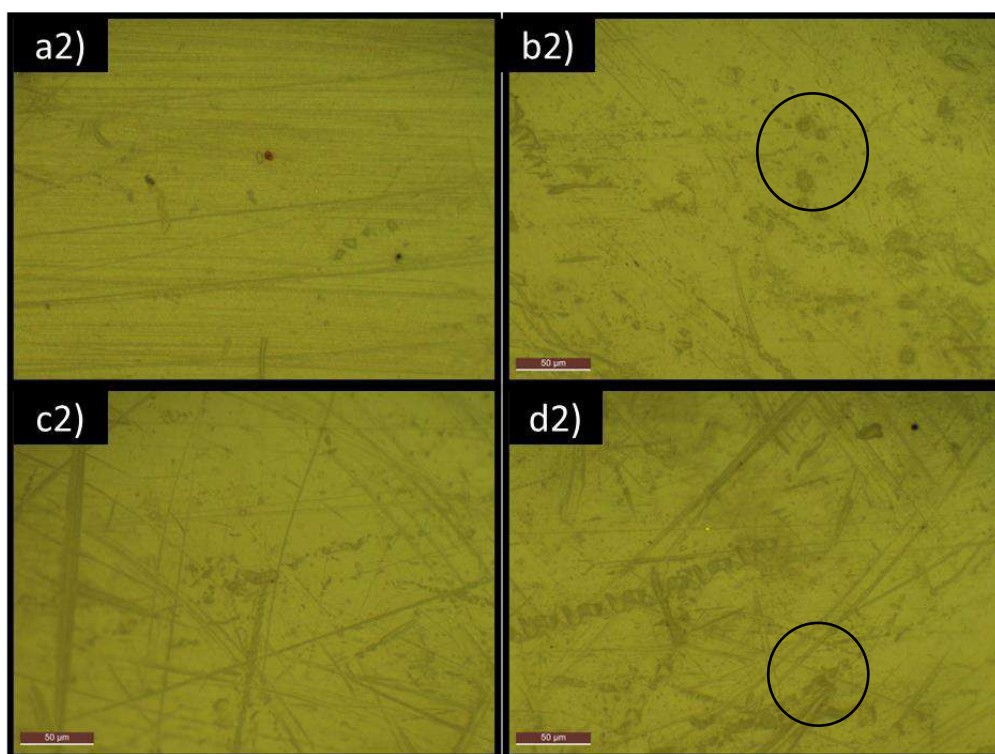
**Figure 4-3** Tensile strength analysis of PES membrane

#### 4.4 OPTICAL MICROSCOPY

Optical microscope uses for analyzing the microstructure of the surface. Here we use OM technique for measuring the microstructure of polyethersulfone membrane. We perform OM at two different ranges, 20  $\mu\text{m}$  and 50  $\mu\text{m}$ . the figures below showing the surface morphology of polyethersulfone membrane at microstructure level. At 20  $\mu\text{m}$  image of OM with 50 resolution reveals that it is dense membrane, dense membrane is our requirement for gas separation. OM results at 50  $\mu\text{m}$  with 20 resolution shows lines and rough structure this occur due to substrate indentation and roughness. Also, in **b2)**, **c2)** and **d2)**, the small circle like structures can be due to water trapped in membrane due to phase inversion method. It may also be due to incomplete evaporation of solvent.



**Figure 4-4** Optical microscopy analysis at 20 micro-meters



**Figure 4-5** Optical microscopy analysis at 50 micro-meters

## 4.5 GAS PERMEATION TECHNIQUE

**Table 4-1** Testing Results of single membranes by Gas permeation setup

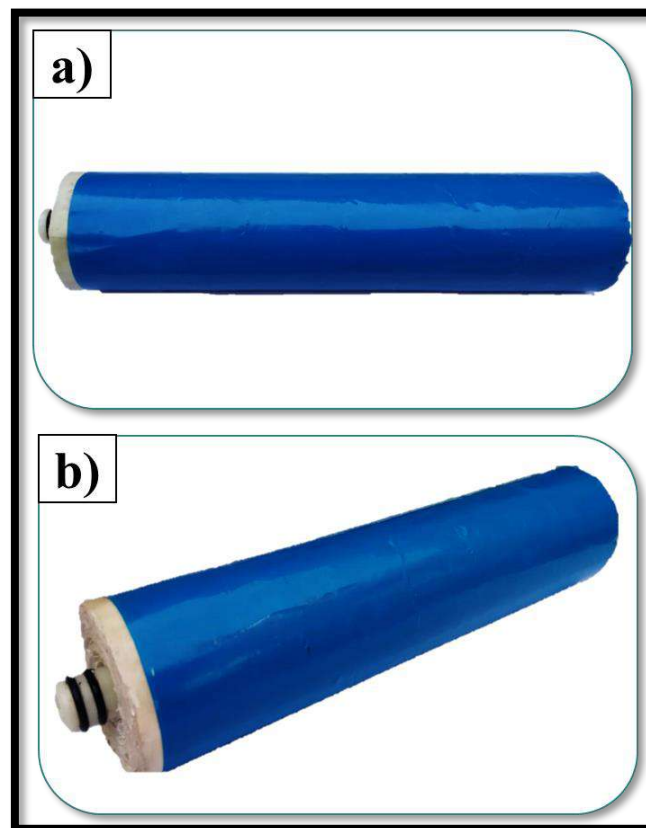
Samples	Thickness (mm)	Permeability (Barrer)		Selectivity CO <sub>2</sub> / N <sub>2</sub>
		CO <sub>2</sub>	N <sub>2</sub>	
Sample 1(a) (with substrate)	210	13.2417	1.7628	7.5116
Sample 1(b) (with substrate)	200	61.1726	9.0698	6.7446
Sample 2 (without substrate)	120	56.3796	8.7744	6.4254

The above results as shown in Table 4-1 shows the permeability and selectivity of three different membranes. Sample 1(a) and (b) are the membranes made by combination of phase inversion method and solvent evaporation method. For these membranes, the temperature of water for dipping was kept about 8-10 °C, whereas, sample 2 was also made by same methods but the difference was the temperature of water for dipping, which was kept as room temperature i.e., 17-20 °C. As we have to make non-porous membranes that's why temperature is a great parameter for controlling pore size and porosity. Low temperature of water reduces the pore size as compare to normal or high temperature. Thus, lower the temperature, lower will be the pores in the membrane and, better permeability and selectivity can be achieved. After dipping the membrane in water, complete solvent evaporation was done by drying in oven, if it is not done accurately then, it has a huge effect on selectivity of membrane.

**Table 4-2** Testing Results of Modules with different configurations

Samples	Diameter (inch)	Permeability (Barrer)		Selectivity CO <sub>2</sub> / N <sub>2</sub>
		CO <sub>2</sub>	N <sub>2</sub>	
Module 1	1.8	56.1519	10.6366	5.2790
Module 2	1.75	13.0426	2.7371	4.7651

Two modules were made with two different configurations. In 1<sup>st</sup> configuration, perforated tube was kept on one side of membrane as shown in above methodology of spiral wound module Figure 3-5, while in 2<sup>nd</sup> configuration which was the complex one, perforated tube was kept in the center of membrane leaf. Both modules were made by two leaves. Both modules should show high selectivity because surface area has been increased by assembling in the form of module, but due to wounding by hand instead of automated wounding equipment, there was a leakage issue in the module. Leakage is controlled by using O-rings and glue. Permeability was achieved by using pressure difference technique of both gases separately. Furthermore, the configuration of module 1 has been used commercially as compare to module 2.



**Figure 4-6** Spiral Wound Module

## **5 CHAPTER 5. CONCLUSSION**

Pore size and porosity can be reduced by temperature in case of pure polymer membrane module. The separation efficiency can also be increased by increasing the leaves of module. This study has established a laboratory-scale spiral wound module with length 12 inch and diameter 1.8 inch respectively by utilizing pure polyethersulfone membrane and the detailed procedure has been described above. Firstly, film was prepared by phase inversion method to produce a dense non-porous membrane and the performance of membrane was investigated by various testing including gas permeation test, optical microscopy, FTIR, UTM and TGA. From the results, it is concluded that the highest selectivity of single membrane achieved in this study was 7.5116, and the highest selectivity achieved by SWM module was 5.2790. It can be further increased by controlling the issue of leakage, which can occur by loose wounding of module. As it may be a human error because wounding was done by hand. Spiral wound module can be made by different configuration and efficiency of spiral wound module depends upon various factors include membrane efficiency, surface area of membrane, configuration at which the module place in separating plant.

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