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Processing, Structure and Properties of Melt Blown Polyetherimide

Kandagor V[1], Prather D[1], Fogle J[1], Bhave R[2] and Bhat G[3]*

[1]*The University of Tennessee, Knoxville, Tennessee, USA*
[2]*University of Georgia, Athens, Georgia, USA*
[3]*Oak Ridge National Laboratory, Oak Ridge, Tennessee, USA*

Abstract

Polyetherimide (PEI), an engineering plastic with very high glass transition temperature and excellent chemical and thermal stability has been processed into membranes of varying pore size, performance, and surface characteristics. A special grade of the polyetherimide was processed by melt blowing to produce microfiber nonwovens suitable as filter media. The resulting microfiber webs were characterized to evaluate their structure and properties. The fiber webs were further modified by hot pressing, a post processing technique, which reduces the pore size in order to improve the barrier properties of the resulting membranes. This ongoing research has shown that polyetherimide can be a good candidate for filter media requiring high temperature and chemical resistance with good mechanical properties.

Keywords: Meltblowing; Microfibers; Polyetherimide; Polymer membranes; Filtration

Introduction

The need for better filter media for applications in liquid and gas filtration has increased in recent years. In particular, filters for biomedical applications, water filtration, oil and gas separation and other applications for reducing environmental pollution have become increasingly necessary. Due to its robust biocompatibility properties, PEI materials are finding applications in the biomedical industry. Recent research has shown that PEI does not exert any considerable level of cytotoxicity or hemolysis, permitting the growth of cell on the surface [1,2]. Membrane filtrations for biological applications have to face two different environments: blood tissues and cell. Consequently, filters for biological and other separation applications must have surface characteristics that are compatible with specific environments that the membranes are intended to be used [1]. With some separation process requiring vigorous procedures which may include high temperature, high pressure, and in some cases high chemical environment, it has become necessary to design filters that can withstand these tough conditions without compromising the effectiveness, quality, and efficiency of the separation process. Nonwoven filters have particularly become attractive because of the simplicity in the fabrication process which does not requires use of solvents that can present challenges during processing as well as in the end product. The PEI resin is a copolymer with the ether molecules between imide groups. The Ultem resin combines the high performance associated with the exotic specialty polymer together with the excellent processability characteristics of engineering polymers, and finds specific applications in the aero, auto, and insulation industries, where performance at high temperatures is a stringent requirement [3].

The melt blowing has become a commercially successful process in producing nonwovens because of its ability to produce fibers of desired characteristics including fiber diameter ranging from 2-4 microns, and desired permeability characteristics, which is achieved by manipulation of the processing conditions. It involves application of hot air jet to an extruding polymer melt, which is then drawn into micro and nano size fibres [4].

The melt blowing technology, which was originally commercialized by the Exxon Chemical Company, is currently widely used for production of fine fiber nonwovens. The extruder melts the polymer and the molten polymer is forced through the melt-blowing die which consists of a row of orifices or jets, resulting in the formation of small diameter fibers [5]. The fibers are then drawn by the high velocity hot air, quenched and collected on a continuous moving belt forming the continuous fiber web. The properties of the meltblown webs are affected by various production parameters including air temperature, polymer/die temperature, die to collector distance (DCD), collector speed, polymer throughput, air throughput, die hole size and air gap [6]. The melt blown fiber webs can be compacted further by calendaring to form membranes with desired surface and pore characteristics.

Several polymers have been successfully meltblown using the pilot line, and many of them are commercially practiced [7,8]. Although ultem has not been melt blown before, it has been converted into fibers and membranes [9]. In this work, we have fabricated flat module membranes from Ultem with high temperature, high chemical resistance, and high pressure operating range and evaluated them for their performance and physical properties [10]. The PEI has been processed by meltblowing and then hot pressed to reduce their pore size at different pressure and temperature for varying structures and properties [11,12] (Figure 1).

Experimental Section

Materials and processing

Commercially available polyetherimide, Ultem 1285, was purchased from Sabic Innovative Plastics, and was used without any further modification. The polymer was dried at a temperature of 130°C for 6 hours to ensure that the moisture content was reduced to the recommended 0.02% before melt blowing. This Ultem had a Melt Flow Rate (MFR) of 23 g/10 min at a temperature of 325°C.

***Corresponding author:** Bhat G, Oak Ridge National Laboratory, Oak Ridge, Tennessee, USA, Tel: +1 865-576-7658; E-mail: gbhat@uga.edu

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Meltblowing was performed using the 15 cm wide meltblowing line (Figure 2) at the University of Tennessee's Nonwoven Research Laboratory (UTNRL). Seven heating zones with independent heaters, including three in the extruder, facilitate incremental heating of the polymer to allow complete melting [13]. The Exxon type die used had 10 holes per cm and each hole was 450 μm in diameter. The die temperature was maintained around 365°C and the air temperature slightly higher (~390°C) to help maintain the die at the desired temperature. Since air pressure is the most critical variable in controlling the fiber diameter, three different air pressures were investigated, keeping rest of the processing conditions same. The primary variable in the production of these fibers was the air pressure because a slight variation in the temperature results in significant change in the permeability of the membrane compared to the variation of the air temperature or collection speed. It has been previously observed that for fibers used in separation technology, the ideal air temperature for Ultem fibers is 390°C. This is the main reason for varying the air pressure (Table 1).

The meltblown Ultem 1285 fiber web was then hot pressed using carver hot press model 3895.4 NE1000 at different temperatures and pressures resulting in further consolidation of the fiber web so that the pore size was reduced to a much lower value that is determined by the pressure and temperatures of the calender rolls. The resulting membrane is bulky and soft on the surface giving it the characteristics of selective permeability.

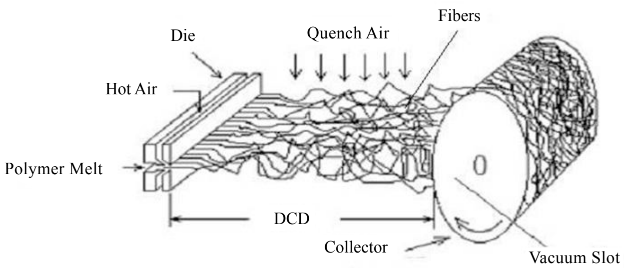

Figure 1: Schematics of the melt blowing process.

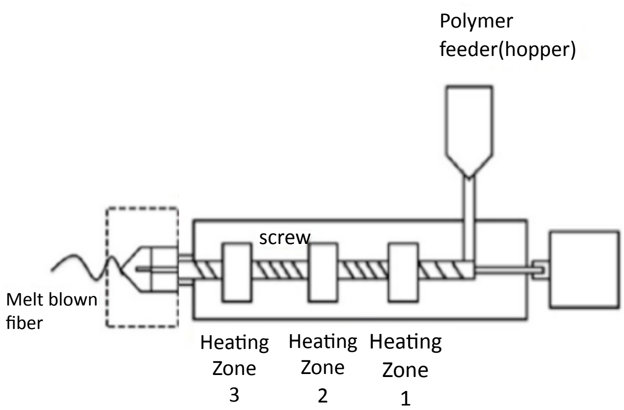

Figure 2: Schematics of the meltblowing line at UTNRL.

	Sample 1	Sample 2	Sample 3
Die temperature	365	365	365
Air Temperature (°C)	390	390	390
Air Pressure KPa	172	206	241
Collector Speed (m/min)	9.3	9.3	9.3
DCD (cm)	12	12	12

Table 1: Summary of the meltblowing conditions for the three samples that were prepared.

Characterization

The produced nonwovens were characterized to determine the pore size and pore size distribution. This was done using a Porous Materials Inc. capillary flow porometer model ASF-1100-AEX. The porometer measures the gas flow as a function of the applied pressure, and the curve that is determined for both the dry and wet measurements is used for the calculation of the pore size, mean flow pore size, the smallest pore size and the gas permeability of the resulting membrane. The Washburn Equation has been used to define the mathematical relationship between the applied pressure and the pore size by using the surface tension and the contact angle of the wetting fluid providing the porosity data [14].

SEM micrographs were obtained using an ETEC Auto-scan scanning electron microscope at 3 KeV after coating with a gold layer. The SEM images used in combination with a computer image processing software (imageJ NIST) helped determine the average fiber diameter and fiber diameter distribution of the samples. The tensile properties of the nonwovens were tested using a United SSEM-1-E-PC tensile tester. Five specimens of each nonwoven sample were cut and the resulting values from the tensile testing were averaged according to the ASTM D638 - 10. The air permeability was measured using TEXTEST FX3300 equipment according to ASTM standard D737-96. The Mettler Differential Scanning Calorimetry (DSC) model DSC821 was used to characterize the polymer meltblown fiber webs and the calendered membranes. The samples were heated at 10°C/min from room temperature to 380°C, held for 3 minutes at this temperature then cooled back to room temperature at 10°C/min [10].

The water flux measurement was performed using an in house water flux measurement set up shown below in Figure 3. The Ultem membrane is subjected to water under pressure and the permeate corresponding to different feed pressure measured. The feed pressure was varied between 13.8 and 68.9 KPa. For experimental accuracy, the time it takes to collect 100 Ml of the permeate was measured for the different feed pressures and the membrane cross flow permeability (LMHB) was calculated for each membrane [15].

Results and Discussions

Fiber diameter

The fiber diameter of melt blown Ultem has been characterized in

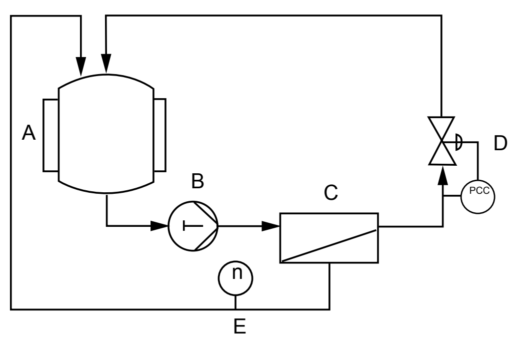

Figure 3: The inhouse membrane flux measurement instrument consisting of: A the vessel, B feed pump, C membrane, D pressure indicator & controller, E permeate flow indicator.

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detail and the correlation between the fiber diameter and the processing air pressure reveals a relationship that is similar to those reported in literature. The lower the air pressure, the higher the resulting fiber diameter and the entangling of the fiberweb. Theoretically, the fiber diameter will increase as the airflow rate decreases [16,17].

Figures 4 and 5 show the fiber diameter and diameter distribution throughout the nonwoven meltblown material, and the data is consistent with the expected results. At the 241 µm, kPa at of 206 pressure, kPa, the average fiber diameter is about 9.5 fiber diameter and increase sat 172 kPa to 10 processing. 5 air pressure, the resulting fiber diameter was determined to be 12 µm. There is a linear relationship between processing air pressure and the resulting fiber diameter as shown in the graph below. The processing air has the most dominant effect on fiber attenuation. In fact, the drawdown of the fiber in the melt blowing process is due to the processing air and higher air pressure leads to acceleration of the molten polymer coming out of the die. That is how the polymer coming out of the die at 450 micron goes down to few microns within a short distance. The higher air pressure means an increase in acceleration of the filament leading to higher velocity and effective draw ratio.

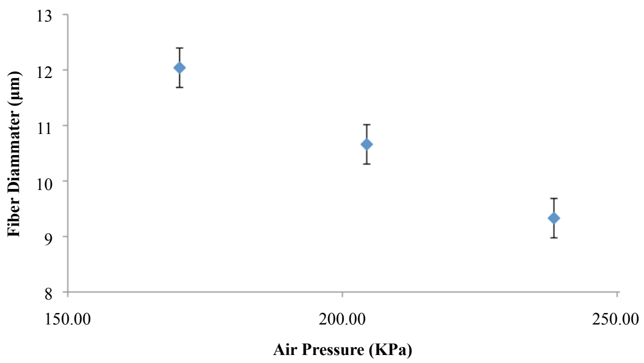

Figure 4: The relationship between the air pressure and the fiber diameter.

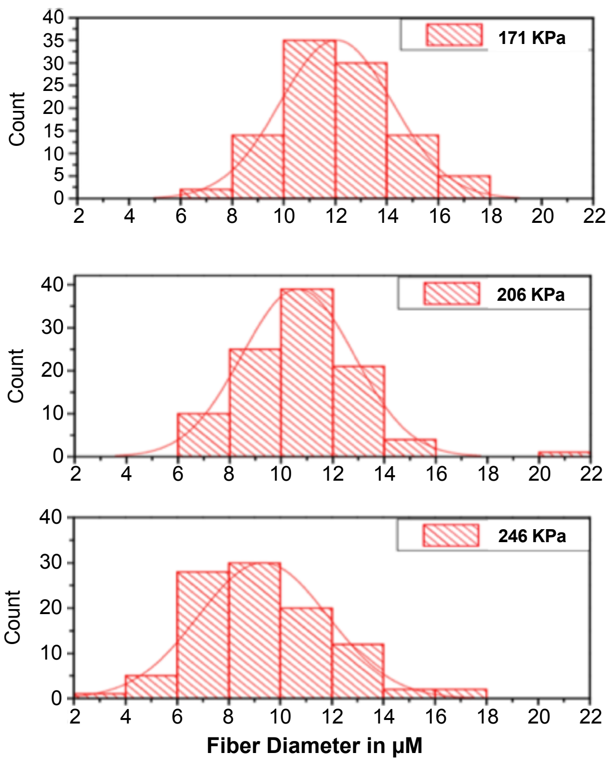

Figure 5: Fiber diameter and diameter distribution.

The fiber diameters are only slightly larger than that of the meltblown webs from typical polypropylenes. Considering the fact that the MFR of the ultem resin was very low and it was not designed to achieve fine fibers, the fiber diameters achieved are very good, especially since the webs were consistently uniform. In fact, the melt blown fibers from many other polymers as well as from earlier PP resins are in the same range. Only because current day commercial PP resins for melt blowing are of special high melt flow rate type, finer fibers in the range of 2-5 microns are possible.

The SEM micrograph of the fiber webs (Figure 6) shows smooth fiber morphology that would confirm the relative ease in the process ability of Ultem 1285 from the pellets by melt blowing, in spite of their high glass transition temperature and melt viscosity. There is no evidence of breaking up of the resulting fibers or variation in diameter along the length of the fiber, an indication of strong fiber web structural integrity. The fiber web formed at higher air pressure shows a lower fiber diameter but higher fiber volume per unit area, which results in a smaller effective pore size and separation characteristics.

Web structure and properties

The thickness of the hot pressed nonwoven fiberwebs decreased with increase in temperature of hot press, and also increasing pressing

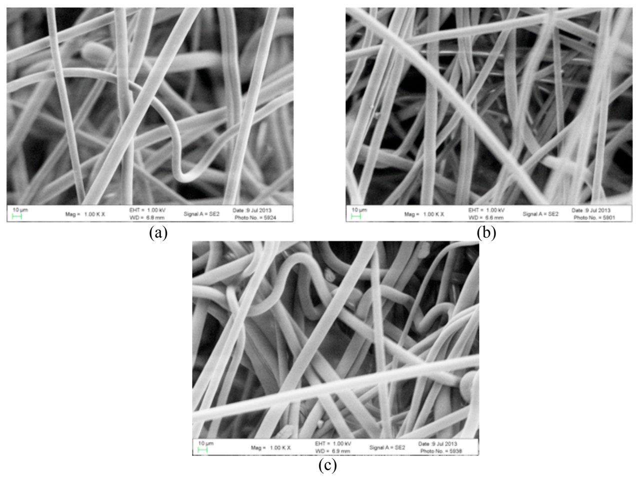

Figure 6: The SEM micrograph of the nonwoven Ultem 1285 fibers and different processing: conditions: (a) 171 KPa, (b) 206 KPa and (c) 246 KPa.

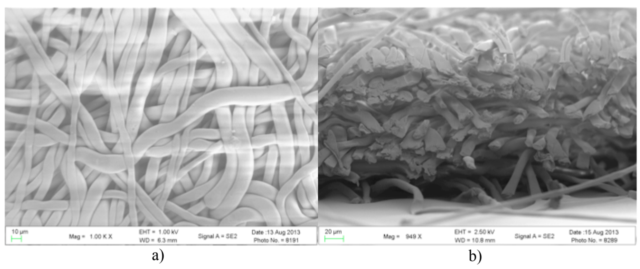

Figure 7: The SEM Micrograph of hot pressed Ultem 1285 fiberweb: a) surface and b) crossectional view.

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time as shown below. The calendering process resulted in the bonding of the web as seen in the SEM micrograph (Figure 7). Comparable observations have been made earlier in structure analysis of calendered webs from various polymeric fibers. This calendering resulted in reduced pore size of the fiberwebs. A combination of shorter time of about 30 seconds and a temperature of 160°C (for the 206 KPa meltblowing condition) resulted in better Ultem membrane as will be seen in the results discussed in following sections [18].

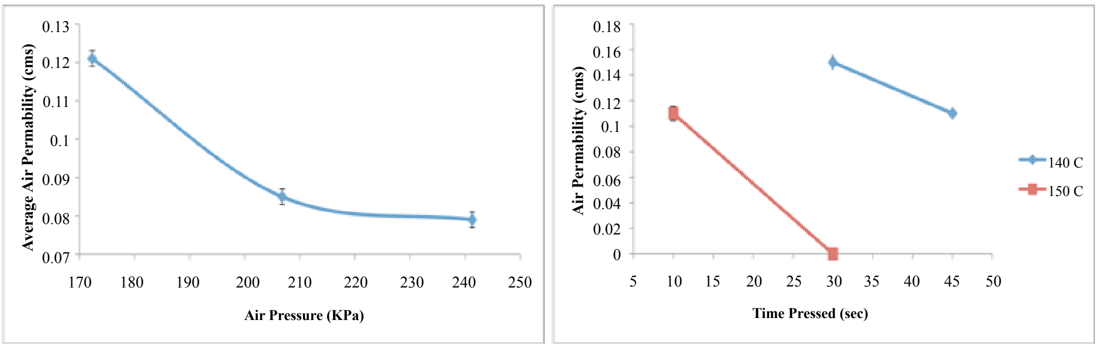
The air permeability of the webs and membranes indicates that the smaller the thickness of the membranes produced after calendering the lower the permeability because of significantly reduced pore size. The membranes that were produced by calendering for longer time at higher temperatures (150°C for 30 sec) resulted in almost zero permeability compared to that which was calendered at lower temperatures for a shorter time as shown in Figure 8.

The change in average thicknesses on calendering. The low meltblowing pressure of 172.4 KPa had the highest thickness and at the pressure of 241 KPa the thickness was reduced membrane thickness, consequently reducing the pore size. This reduction in thickness due to the combined effect of heat and pressure, which increases the packing density of the fibers shown in Figure 9.

The relationship between the meltblowing pressure and the average mean flow pore diameter (MFPD) of the membrane is unique. The MFPD is that defined as value for which the flow in the membrane is reduced by half in a partial flow test in a capillary flow parameter instrument. Although at lower meltblowing pressure the fiber diameter is larger and increases with the increasing pressure as shown earlier, it is the fiber density that will determine the MFPD. The graph in Figure 10 shows that at the meltblowing air pressure of 206 KPa, the fiber diameter is smaller than at 172 KPa air pressure, but larger than the 241 KPa processing pressure. However the pore size and pore size distribution are the results of fiber diameter as well as fiber packing. In general finer fiber diameter results in smaller pore size. The pore structure is further reduced when calendering is done at different temperatures due to more compact packing. Thus pore size can be manipulated by changing the temperature and pressure to achieve a desired pore structure.

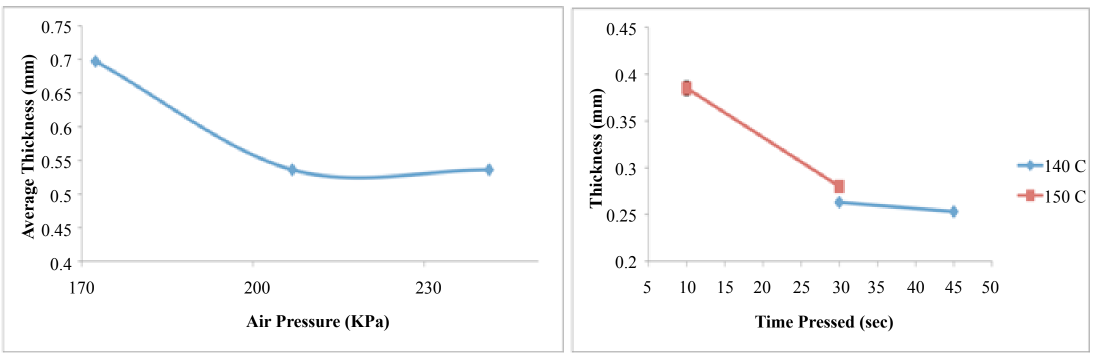
Tensile properties

Reproducible data shows that the tensile strength of the nonwoven Ultem 1285 produced at the pressure of 241 KPa averaging 8 KN/m^2. The peak elongation for this sample was 11.1%. The tensile strength is a combined effect of fiber diameter, web consolidation as well as total mass per unit area of the fabric. The results for the fibers produced at 206 KPa and 172 KPa meltblowing pressure are relatively same, but the values are higher compared to that produced at 241 KPa air pressure as shown in Figure 11. This means that the strength of the webs is almost similar and as the meltblowing pressure decreases, the increase in the strength is not larger. The peak force for these fibers is about 12.7 KPa and an average elongation of about 13% as shown in Figure 11. The breaking load increased with increasing die air pressure that could be due to the increase in basis weight and thickness. The elongation decreased with increasing die air pressure in the production direction, due to increase in the breaking load. The breaking load increased due to stronger bounding of the fibres in the web as a result of increasing air pressure applied to the web by the vacuum [19,20].



a) b)

Figure 8: The relationship betweeen the a) calendring time/membrane air permenace and b) the meltblowing pressure/membrane air permeance



a) b)

Figure 9: The relationship between the thickness of the resulting Ultem 1285 meltblown webs: a) meltblowing pressure, and b) the calendering temperature/time.

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Thermal analysis

The glass transition temperature of the Ultem 1285 pellets and the meltblown webs obtained from different processing conditions was the same and measured at 177.62°C for heating and average of 185.2°C for the cooling process. No significant difference in morphology was observed for the different melt blown samples as shown in Figure 12. Ultem being an amorphous polymer does not show any melting peaks and only change in slope around the glass transition temperature. Accordingly, we observed the glass transition temperature in all the nonwoven samples at temperature close to that of the original polymer, only a few degrees lower due to the kinetic effect of the process as the finer fiber samples show better heat transfer due to higher surface area [21].

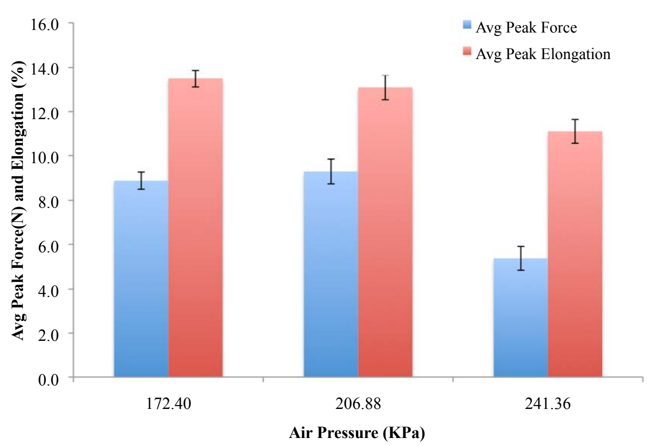

Figure 10: The graphical representation of the meltblowing pressure to the mean flow pore diameter.

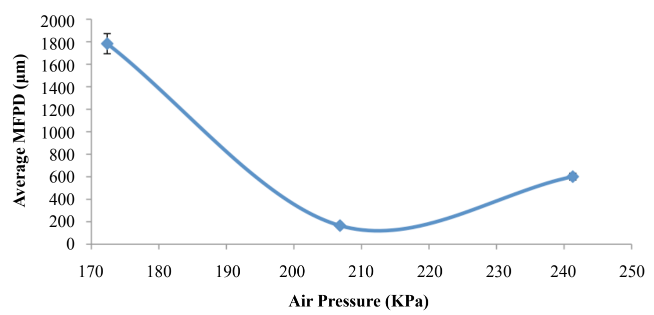

Figure 11: Tenisle strength and elongation of meltblown webs produced at different air pressures.

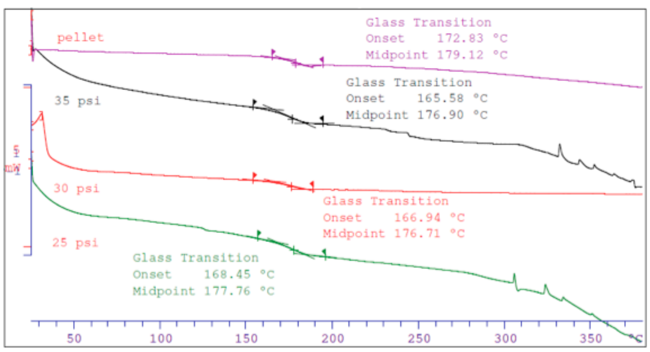

Figure 12: DSC heating curve for Ultem 1285 pellets and fiberweb produced under different processing.

Liquid flux

All the membranes that were tested were produced at 241 KPa meltblowing pressure and hot pressed for 10 seconds at different temperature. The Ultem 1285 membrane calendered at 150°C reduced the LMHB by almost 50% compared and the membranes produced by calendering at 160°C and 175°C had a LMBH reduction of about 90%. This means that at higher calendering temperatures, the membrane pores are not reduced significantly and the permeation characteristics are almost similar. The ideal calendering condition is therefore 160°C for 10 seconds. The pore size can also be manipulated to a specified conditions depending on the desired separation characteristics by changing the calendering conditions (Figure 13) [15].

The separation characteristics of the membrane formed from meltblown Ultem 1285 can be used for different separation application at high temperature and high pressure. The increase in filtration efficiency of the membrane was mainly due to physical changes in porosity and permeability, as the surface of the membrane did not acquire any charge [22,23].

Conclusions

In this work, we have fabricated fibrous membranes by meltblowing Ultem, which is a high temperature stable, high performance polyetherimide, and investigated the structure and properties of the resulting meltblown web membranes. The microfibers were produced at different meltblowing air pressures and calendered in order to reduce the pore size of the membrane by varying the calendering temperature, pressure, and time. The fiber diameters varied by 8 to 13 microns depending on the air pressure used, and there were differences in air permeability as well as pore size of these webs as expected from differences in fiber diameters. It was established that the separation characteristics of the resulting membrane can be changed by varying the calendering parameters that determine the pore size of the membrane, and therefore its permeability. Membranes that were calendered between 165°C and 175°C, had the same pore size while the membrane formed by calendering the fiber web at 150°C had slightly larger pores. Their separation characteristics varied by about 20,000 liters of water per meters squared per hour per bar (LMHB). This is a significant improvement in the mechanical filtration quality of the membrane. It was clearly demonstrated that the filtration

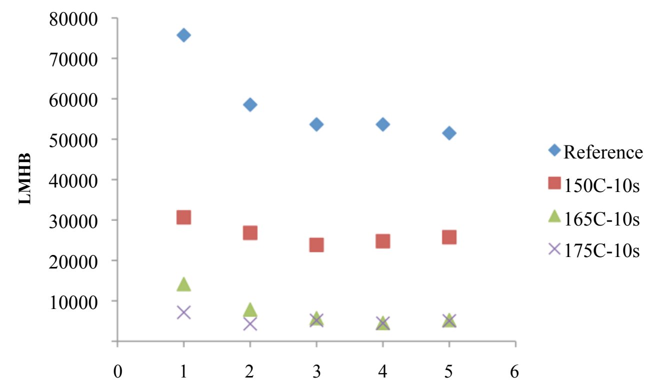

Figure 13: Calculated LMHB for each of the Ultem membrane fabricated at different condition ns.

characteristics of the membranes formed from this high temperature polymer can be tailored by manipulating different meltblowing and calendering parameters to produce membranes with desired pore size for a specific filtration quality.

Acknowledgments

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