Effect of extrusion rate on morphology of Kaolin/PolyEtherSulfone (PESf) membrane precursor

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Abstract. This study aims to investigate the influence of apparent viscosity induced by spinneret geometry and extrusion rate on morphology of Kaolin/PESf hollow fiber membranes. Different extrusion rates at two different rheology properties were introduced on a straight and conical spinneret resulting in various shear rates. The hollow fiber membrane precursors were spun using the wet spinning method to decouple the effect of shear and elongation stress due to gravity stretched drawing. The morphology of the spun hollow fiber was observed under Scanning Electron Microscope (SEM) and the overall porosity were measured using mercury intrusion porosimeter. Shear rate and apparent viscosity at the tip of the spinneret annulus were simulated using a computational fluid dynamics package; solidworks floworks. Simulation data shows that extrusion rate increment increases the shear rate at the spinneret wall which in turn reduce the apparent viscosity; consistent with a non Newtonian shear thinning fluid behavior. Thus, the outer finger-like region grows as the shear rate increases. Also, overall porosity of hollow fiber membrane decreases with extrusion rate increment which is caused by better molecular orientation; resulting in denser hollow fiber membrane. Thin outer finger-like region is achieved at low shear experience of 109.55 s⁻¹ via a straight spinneret. Increasing the extrusion rate; thus shear rate will cause outer finger-like region growth which is not desirable in a separation process.

1. Introduction

Membrane technology have benefitted a large swath of industry ranging from water treatment, gas separation and chemical engineering. With the discovery of the first asymmetric cellulose membrane by Leobe and Sourirajan, 1963 [1], the technology has developed and matured thru all the years. However, new application and challenges demands better features such as higher temperature tolerance and chemical resistivity. Thus, research has been focused on exploring inorganic materials as alternative membrane constituents. Example of inorganic are such as carbon or ceramic based material. Ceramic membrane have the advantage of water resistant, thermally stable, chemically resistant and good wear properties [2]. Example of such ceramics are as; γ-alumina, zirconia, titania and silica [3]. In an effort to find alternative cheaper ceramic membrane, Kaolin has been explored as an alternative ingredient for hollow fiber membrane [4].

Membrane products comes in different form or configurations; flat sheet, spiral wound and hollow fiber tubes. Hollow fiber tubes are in particular interest as it has a high surface area to volume ratio. Spinning technique based on the phase inversion process used in fabrication of hollow fiber membrane

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is a complex process and encompass a number of spinning variables. The membrane morphology, mechanical properties and performance are governed by spinning conditions such as the shear rate [5], bore fluid rate [6] air gap [7], spinneret design, material properties [8, 9], viscosity and other factors.

In a spinning process, a viscous dope solution is extruded through the annulus of a spinneret. Given the small size of the passage ways and the annulus; coupled with non Newtonian behavior of the dope solution. It is subjected to shear stress and molecular orientation which researchers have ascertained that the rheological behavior of the suspension and spinning variables affect the final morphologies and performance of hollow fiber membrane [10, 5, 11,12]. The particles of the fluid move relative to each other so that they have different velocities, causing the original shape of the fluid become distorted [13]. Therefore, it is vital to study the fluid behavior and its correlation with spun hollow fibers performance.

Computational fluid dynamics (CFD) technology makes it possible to study the intrinsic value of a fluid behavior during spinning process. The methodology has been used extensively in prior research to understand and established correlation between fluid flow profiles and hollow fiber performance [14,15]. However, literature study has showed that current study of shear rate and elongation rate effect on membrane performance are dominated by organic membrane study.

The objective of this paper is to study the effect of extrusion rates induced in a spinneret on Kaolin/PESf hollow fiber characteristic. To achieve this objective, the following sub objective were completed as well; (1) Spinning of hollow fibre precursor; (2) CFD study of shear rate and apparent viscosity profile; (3) Hollow fiber morphology and porosity study of hollow fiber precursor.

2. Methodology

The investigative work is carried out in three phase; preparation of the kaolin suspension, spinning and characterization of hollow fiber precursor and simulation of hollow fiber spinning process using cfd method. The Kaolin suspension preparation includes rheology assessments which provides necessary data for simulation work while characterization of the spun hollow fiber membrane gave insights of the hollow fiber performance. Finally, simulation works were made to study the suspension flow behavior inside the annulus which is correlated with hollow fiber morphology and porosity. The correlation of fluid flow behavior and hollow fiber characteristic would enable researchers to predict hollow fiber performance in the design phase thus reduce development time and cost.

2.1. Kaolin solution

Kaolin powders, polyethersulfone (PESF) in pellet form and N-methyl-2-pyrrolidone (NMP, 99% extra pure) were purchased from Sigma Aldrich; each were used as a binder and solvent. The dope solution was prepared as a procedure published elsewhere (Suffian et al., 2014), whose rheological properties were determined by using Brookfield’s Programmable HADV-IV + Rheometer. Two type of dope solution with different Kaolin ratio were used as shown in table 1.

| Properties                  | A    | B    |
|-----------------------------|------|------|
| Kaolin                      | 27 g | 54 g |
| PESf                        | 27 g | 27 g |
| Consistency factor, K (Pa.s)| 1.9953 | 2.5527 |
| Flow Index (n)              | 0.7  | 0.8  |

The suspension obeys the power law model commonly associated with the shear thinning non Newtonian fluid behaviour (Suffian et al., 2014). A power law model is normally presented with Eq. 1.
2.2. Spinning of hollow fiber precursor
Hollow fiber precursor was prepared through a wet spinning process (Chun et al., 2004). The formulated dope solution is extruded through a tube-in-orifice spinneret under a pressurized nitrogen gas and exited directly on the coagulation bath. Such measure isolates the effect of elongation rate due to gravity and solvent evaporation that normally took place in a dry-wet spinning system with an air gap. Two type of spinneret were used; straight and conical spinneret. Tap water at 25 °C was used both as the external coagulant agent in the coagulation bath and the bore fluid at constant flow rate. The spun fibers were rinsed with water at room temperature for at least 24 hours to remove solvent residue. The process is then repeated for each different extrusion rate and the coagulant (water) was renewed for each spinning process. Sample designation is shown in Table 2.

Table 2. Sample designation.

| Spinneret Type | Sample Designation | Suspension Type | Extrusion Pressure Abs. (bar) |
|----------------|--------------------|----------------|-----------------------------|
| Straight       | S1                 | A-Kaolin/PESf: 1.0 | 1.3                         |
|                | S2                 |                | 1.4                         |
|                | S3                 |                | 1.5                         |
|                | S4                 | B-Kaolin/PESf: 2.0 | 1.3                         |
|                | S5                 |                | 1.4                         |
|                | S6                 |                | 1.5                         |
| Conical        | S7                 | A-Kaolin/PESf: 1.0 | 1.1                         |
|                | S8                 |                | 1.2                         |
|                | S9                 |                | 1.3                         |
|                | S10                | B-Kaolin/PESf: 2.0 | 1.1                         |
|                | S11                |                | 1.2                         |
|                | S12                |                | 1.3                         |

The hollow fibers pre-cursor morphology were observed under Scanning Electron Microscope (SEM). The preparation procedures are as follow; the fibers are first dried to ensure liquid are removed from the specimen. Then, it is quenched in a liquid nitrogen solution, which brought the specimen to cryogenic state. Next, a thin layer of Joel JFC-11-E is sputtered on the fiber membrane using a sputtering device. Finally, images of the hollow fiber membrane were taken at various magnification size.

The pore size and porosity of the prepared hollow fiber pre-cursor were measured with a mercury porosimeter (Thermoscientific Pascal 440) which uses the mercury intrusion technique. In this technique, mercury is forced in to a dry membrane at incremental pressure. The corresponding mercury volume at each pressure is determined and calculated by the instrument software. A sample is first prepared for the measurement by drying in an oven at 40 °C for 24 hours. Then, it is transferred onto a dilatometer and mercury is added in to the chamber. Then it is mounted on the porosimeter and the pressure intrusion process is initiated. Measurement data is recorded after the intrusion process is complete.
2.3. Simulation work
Solidworks floworks software were used to simulate the flow profile inside the spinneret annulus. The design geometry of the straight and conical annulus spinneret used to spin the hollow fiber membrane are shown in figure 1 which are simplified model of the actual design to save computation resources. Similar spinning parameters reflecting the actual spinning process were made which totalled 12 samples. The properties of interest in the fluid flow region would be the shear rate and velocity profile at the tip of the annulus. In this outer most region, the kaolin suspension immediately exposed to coagulation agent in the bath where the morphology of the hollow fiber took form. The following assumptions were made during the simulation process; (1) Fully develop flow (2) Steady state condition (3) Homogeneous suspension (4) Atmospheric pressure at outlet (5) No slip boundary condition.

![Figure 1. Design geometry of Spinneret.](image)

2.3.1. Governing equation
Computational fluid dynamics uses numerical techniques to solve fluid flow equations and demonstrate them in much more presentable manner. Solidworks flow simulation solve the Navier-Stokes equation which involve the conservation law for mass, angular momentum and energy which are presented in the Cartesian coordinate system rotating with angular velocity about an axis pass through the origin coordinate system can be written as follow [16]:

\[
\frac{\partial \rho}{\partial t} + \frac{\partial}{\partial x_i} \left( \rho u_i \right) = 0
\]  

(2)

\[
\frac{\partial \rho u_i}{\partial t} + \frac{\partial}{\partial x_j} \left( \rho u_i u_j \right) + \frac{\partial}{\partial x_i} \left( \tau_{ij} + \tau^R_{ij} \right) + S_i = 0
\]  

(3)
The working fluid in this study exhibit a Non-newtonian power law behavior as found by Suffian et. al., 2014. The power law model described as follows (Solidworks Inc, 2011):

\[ \mu(\dot{\gamma}) = K(\dot{\gamma})^{n-1} + \frac{\tau_o}{\dot{\gamma}} \]  

Where \( n < 1, \tau_o = 0 \) describes the power law model of shear-thinning non-Newtonian liquids.

2.3.2. Grid independence study

A grid independence test was done to ensure that a good balance of solution quality and processing resources achieved. In this study, both straight and conical spinneret were analyzed with different mesh densities. The meshes statistic are as shown in Table 3. Figure 2 and Figure 3 compares the velocity profile variance at the tip of the annulus.

| Geometry         | Designation       | Cell Type | No. of Elements |
|------------------|-------------------|-----------|-----------------|
| Straight Spinneret | Straight A (Coarse) | Fluid     | 75524           |
|                   |                   | Partial   | 39646           |
|                   | Straight B        | Fluid     | 103192          |
|                   |                   | Partial   | 46294           |
|                   | Straight C        | Fluid     | 158634          |
|                   |                   | Partial   | 61168           |
|                   | Straight D (Fine) | Fluid     | 277756          |
|                   |                   | Partial   | 80764           |
| Conical Spinneret | Conical A (Coarse)| Fluid     | 27597           |
|                   |                   | Partial   | 13030           |
|                   | Conical B         | Fluid     | 1461481         |
|                   |                   | Partial   | 150858          |
|                   | Conical C (Fine)  | Fluid     | 1804974         |
|                   |                   | Partial   | 181122          |

As the mesh density increases, the variance between velocities of each density become smaller. However, each mesh density increment also causes the processing time to increase thus a balance between processing time and mesh dependence is sought. The smallest maximum variance of velocity between mesh density is used to determine which mesh density is to be used for the rest of the simulation. For straight spinneret, maximum variance of velocity for Straight C, and Straight D is tolerable (+/- 0.001). Thus, mesh density – Straight C is used as the mesh configuration in CFD study for straight spinneret. As for conical spinneret, maximum variance observed in Conical C at 0.001 and thus the mesh statistic is used in the simulation work.
Figure 2. Velocity profile at the tip of the annulus for straight spinneret.

Figure 3. Pressure profile along axial axis at inner tube surface for conical spinneret.
3. Result and discussion

3.1. Hollow fiber morphology

The morphology of the hollow fiber precursors was observed under Scanning Electron Microscope (SEM) imaging system. The spun fibers demonstrated axisymmetric structure; a sponge like structure in the center, finger like structure with narrow long voids originating from the inner and outer surface of the hollow fiber membrane as shown in Figure 4. The finger like structure attributes were caused by rapid precipitation near the surface of the membrane while a slower precipitation caused sponge like structure which is normally in the middle section of the hollow fiber [17,18,19].

![Figure 4. Hollow fiber precursor (S7).](image)

Figure 5 shows regions growth for samples 1 to 6 spun using straight annulus. The x-axis shows different region position in the hollow fiber membrane morphology, and the Y-axis shows area ratio of each type of region. The dots represent area ratio at different region position for each samples and summation of each point return 100%. From Figure 5, it can be seen that inner region dominates the fiber cross section. The domination of the inner region indicates that a fast precipitation occurs in the inner surface for both suspension types. Concentration of kaolin particles plays an important role as different amount of coagulant between inner and outer coagulant agent affect precipitation rate. As the extrusion rate increases, the inner region tends to shrink while outer region tends to grow which indicates faster extrusion rates exposes Kaolin suspension to larger volume of coagulant in the outer region; promoting faster precipitation.
Figure 5. Region growth for sample S1, S2, S3, S4, S5 and S6.

Finger-like region growth in the conical spinneret are also observed and examine detail. Figure 6 shows region analysis for sample S7 to S12. As oppose to region development observe in a straight spinneret, the outer region dominates the hollow fiber microstructure. It can be seen from Figure 4, the outer region dominates and continue to doing so with extrusion rate increment while inner region decreases. Similar trend to lower Kaolin ratio is observe for outer region and inner region growth, where the outer region tends to increase while the inner region tends to decrease. Interestingly, the outer and center region in the conical spinneret tends to be more dominance compared to a straight spinneret.

Figure 6. Region growth for sample S7, S8, S9, S10, S11 and S12.

3.2. Effect of extrusion rates on hollow fiber precursor porosity
Mercury intrusion porosimeter was used to calculate the average pore diameter and overall porosity. Table 4 shows the measured average pore size and overall porosity. The porosity of the membrane precursor ranges from 78% to 23% with the trend shows decreasing porosity and average pore
diameter as extrusion rates increases. Literature have shown that membrane porosity ranges around 20% to 90% which put the membrane precursor in this study in acceptable range [20, 21].

Table 4. Measured average pore diameter (μm) and overall porosity (%) for hollow fiber precursor.

| Spinneret Type | Sample Designation | Suspension Type | Extrusion Pressure (bar) | Avg. pore dia. (μm) | porosity (%) |
|----------------|--------------------|----------------|--------------------------|---------------------|--------------|
| Straight       | S1                 | A- Kaolin/PESf: 1.0 | 1.3                     | 0.2589               | 76.00        |
|                | S2                 |                | 1.4                     | 0.2014               | 53.20        |
|                | S3                 |                | 1.5                     | 0.1339               | 31.15        |
|                | S4                 | B- Kaolin/PESf: 2.0 | 1.3                     | 0.2244               | 68.25        |
|                | S5                 |                | 1.4                     | 0.1219               | 48.15        |
|                | S6                 |                | 1.5                     | 0.1157               | 23.61        |
| Conical        | S7                 | A- Kaolin/PESf: 1.0 | 1.1                     | 0.2364               | 78.17        |
|                | S8                 |                | 1.2                     | 0.1459               | 59.43        |
|                | S9                 |                | 1.3                     | 0.1514               | 48.30        |
|                | S10                | B- Kaolin/PESf: 2.0 | 1.1                     | 0.1539               | 72.61        |
|                | S11                |                | 1.2                     | 0.1313               | 42.13        |
|                | S12                |                | 1.3                     | 0.1272               | 30.32        |

Similarly, higher Kaolin content also causes lower porosity compared to lower kaolin content. This shows that higher Kaolin content increases the concentration of Kaolin and the density of the precursor which in turn reducing the porosity. This can be explained by the increase of viscosity due to increase in Kaolin ratio as shown in section x. The increase of the solution viscosity delays the Kaolin suspension-solvent demixing as the solvent-non solvent exchange rate is slowed down. Thus a dense skin layer is formed before demixing occurs which depends upon the extent of demixing process slowdown [22,23].

It appears that the porosity and average pore diameter of the hollow fiber precursor is highly affected by the extrusion rates. The porosity of the precursor membrane has shown decreasing trend both for different Kaolin ratio and different design of spinneret as extrusion rate increases. Qin and Chung, 1999 [11] have reported that higher extrusion rates would cause smaller pore size and a denser skin due to molecular orientation. They postulate, a molecular chain that experienced high shear rate aligns themselves causing the molecules to pack closer together during the demixing process forming a tight structure. Overall porosity of a membrane and the average pore diameter can give insights of the membrane potential use. It is known that a membrane with a high porosity will have good permeability while a low porosity have’s good selectivity [24].

3.3. Shear Rate Profile of Dope Solution by CFD Simulation

Table 5 shows a quantitative value of the axial velocity at the outlet of the straight spinneret for both Kaolin/PESf ratio 1 and 2 (A and B). The shear rate records maximum value near the wall of the spinneret and exhibits minimum value at the center of the convex. There is no clear deviation between Kaolin/PESf ratio in terms of dope solution axial velocity and shear rate.
Table 5. Axial velocity and shear rate statistics for straight spinnerets.

| Sample | Max. Axial velocity (m/s) | Inner wall Shear Rate (1/s) | Outer wall shear Rate (1/s) |
|--------|---------------------------|-----------------------------|-----------------------------|
| S1     | 0.06388                   | 448.24                      | 347.77                      |
| S2     | 0.09939                   | 697.46                      | 541.13                      |
| S3     | 0.13976                   | 980.68                      | 760.87                      |
| S4     | 0.02151                   | 138.78                      | 109.55                      |
| S5     | 0.03067                   | 197.87                      | 156.19                      |
| S6     | 0.04162                   | 268.45                      | 211.90                      |
| S7     | 0.0872                    | 626.65                      | 471.12                      |
| S8     | 0.2316                    | 1663.31                     | 1250.42                     |
| S9     | 0.3973                    | 2706.23                     | 2155.78                     |
| S10    | 0.0297                    | 188.49                      | 151.01                      |
| S11    | 0.0703                    | 445.69                      | 357.09                      |
| S12    | 0.1194                    | 757.36                      | 606.78                      |

3.4. Correlation of extrusion rate to hollow fiber morphology

Relation of the shear rate at the outer surface of the hollow fiber and finger like region development at the outer layer of the hollow fiber membrane are shown in Figure 7, Figure 8, Figure 9 and Figure 10. As the extrusion rate increase, the shear rate at the wall increases albeit at different rate depending on type of spinneret and suspension. The shear rate observed in conical annulus tends to be higher compared to straight annulus. Suspension type B which is more viscous due to higher Kaolin loading tends to have lower shear rate compared to suspension type A. Observing the outer region development, shear rate increment caused the outer finger like region to develop; regardless of annulus type and suspension.

Figure 7. Shear rate and outer region development for sample S1, S2 and S3.
Figure 8. Shear rate and outer region development for sample S4, S5 and S6.

Figure 9. Shear rate and outer region development for sample S7, S8 and S9.
The morphology response to shear rate is believed to be associated with the viscosity of the Kaolin suspension itself. It was determined that the Kaolin suspension exhibits a shear thinning (Pseudo-plastic) non-Newtonian fluid behaviour [25]. As the shear rate increases at the outer surface of the spinneret annulus, the apparent viscosity decreases which affects the precipitation mechanism in the outer surface of the hollow fiber precursor.

Kingsbury and Li, 2009 [19] postulates that high viscosity inhibits precipitation rate and growth of finger like voids at the surface of hollow fiber membrane. Thus it is possible with the decrease of viscosity at the outer surface of hollow membrane, encourage faster precipitation rate at the outer surface of the hollow fiber membrane. To boot, faster extrusion rates exposes the suspension to larger coagulation agent in the coagulation bath causing faster precipitation rate.

4. Conclusion
This research attempts to produce an assymetrical Kaolin/PESf hollow fibre membrane by applying computational fluid dynamics (CFD) method to develop correlation between extrusion rates and hollow fiber morphology. For this purpose, the research work is carried out in phases; a) Spinning and characterization of Kaolin hollow fibre membrane, b) CFD study, and c) Correlation of hollow fibre morphology to CFD data.

The shear rate value at the inner and outer wall obtain from CFD simulation is compared to inner and outer region growth. There is a strong correlation between region growth and shear rate on the annulus wall; outer region growth and inner region reduction against shear rate increment.

An interesting observation were made in this study, although it is clear that shear rate has an obvious effect on morphology of the hollow fibre membrane but other contributing factor are also found; the shape of the annulus. A conical annulus affects the fluid flow to an extent where a common correlation between a straight and conical spinneret to be established. In the straight spinneret, the Kaolin suspension enters the small annulus and able to attain fully develop flow. However, the angled shape of the conical annulus means that the Kaolin suspension is not fully develop flow and experience higher shear rate. Considering that the kaolin solution is a non Newtonian fluid obeying the
power law scheme, this study concludes that an increase in shear experience reduce the apparent viscosity and ultimately increases the precipitation rate. Thus, explains dominance of the outer finger-like region as the extrusion rate increases.

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