CHAPTER 3

MATERIALS AND METHOD

3.1 Materials and Chemicals

Chemicals use in this study is Poly (vinylideneflouride) (PVDF) polymer pellets was purchased from Sigma Aldrich, N-methyl-2-pyrrolidone (NMP) with 99.5% purity and Lithium Chloride (LiCl) were purchased from Sigma Aldrich. The CO2 and N2 gases were prepared by Chemical Engineering Laboratory, Universiti Malaysia Pahang.

3.2 Methods

The method use for this study is the dry/wet phase inversion method. Phase inversion process is the process where the phase separation undergoes the induction in the homogenous polymer solution. There are three conditions for the induction in phase inversion process which is either by temperature change or by wet process where the solution is immersed in a nonsolvent bath or by dry process where the solution can also be exposed to the nonsolvent atmosphere. The thermal process usually will be applied for a polymer with poor solubility like propylene (Nunes & Peinemann, 2006; Khulbe et al, 2008).

However, for the case of producing PVDF membrane which is a group of hydrophobic thermoplastic polymers that have a stability of fluoropolymers with –CH2 and –CF2 groups of polymer chains that will give a polarity which enable polymers to be dissolved in certain solvents. Its selective solubility has become an advantage for PVDF membranes to be fabricated using phase inversion technique. The phase inversion process is better to use the dry phase inversion and wet phase inversion process to get a porous type membrane and the morphology is asymmetric. As for this study, the process used is dry/wet phase inversion (Sun et al, 2012; Nunes & Peinemann, 2006).

Dry/wet phase inversion process requires a polymer solution to be prepared first by mixing the polymer with a solvent. Then, the polymer solution is cast on an appropriate surface such as glass plate using a hand-casting knife to the required thickness. A partial evaporation of the solvent takes place and the cast film is then immersed in a non-solvent medium. Immersion into the non-solvent medium creates a solvent-nonsolvent exchange that brings a thermodynamically stable system that cause two phases coexist. A thin layer of dense polymer is formed at the top of cast film during the first step of desolvation where the solvent evaporate. The poor-polymer phase will give an increment of the pores (Nunes & Peinemann, 2006; Khulbe et al, 2008).

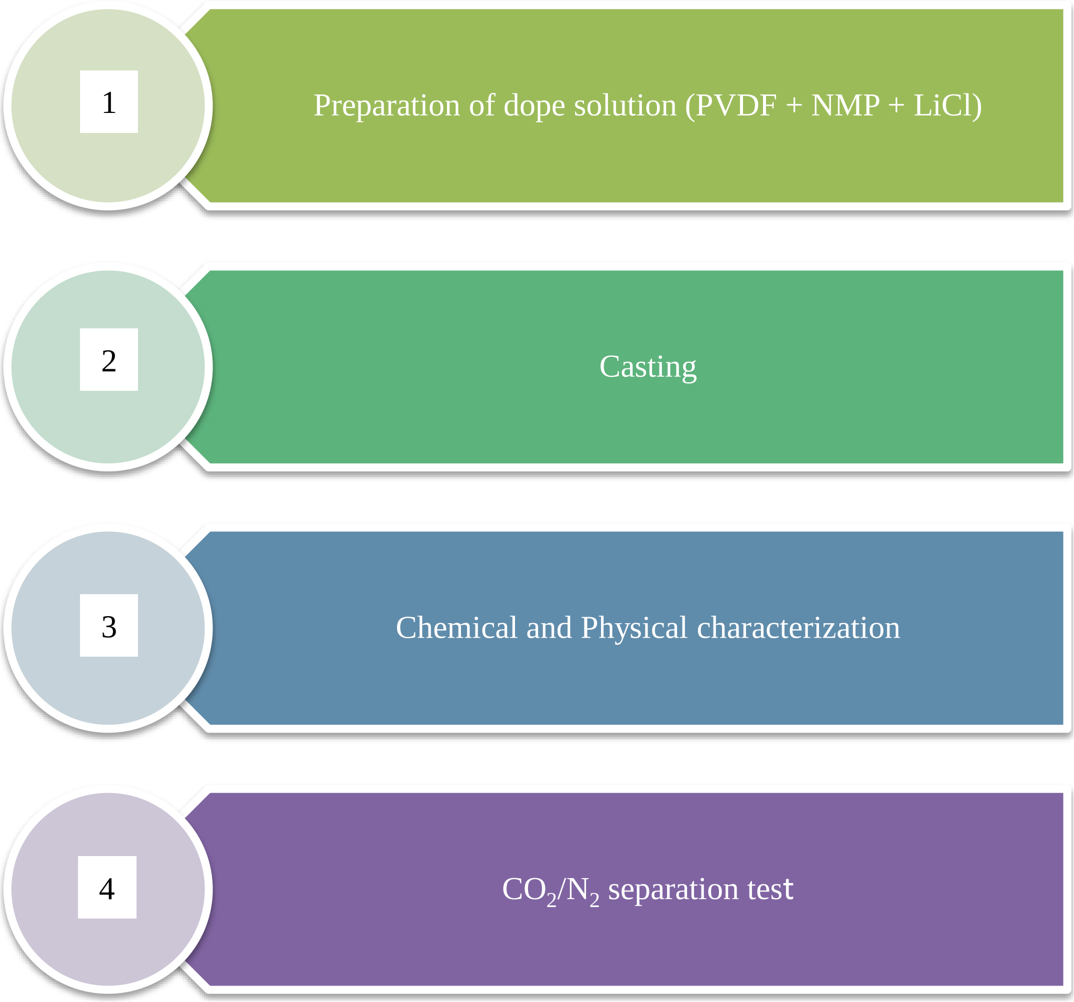

Figure 3-1: Methods of production of PVDF membrane for CO_2/N_2 separation

3.2.1 Preparation of casting solution

The casting solution was produced by mixed PVDF polymer and NMP together with LiCl as an additive for this study. Four casting solutions were prepared as the ratio of the mixing compound is manipulated such that (PVDF polymer:NMP: LiCl) as (17:83), (16:83:1), (15.5:83:1.5), (15:83:2). The casting solutions that consists of the mixture of PVDF polymer and NMP were stirred at temperature 60°C by using magnetic stirrer for about ten hours. Then, appropriate amount of LiCl additive was added to the casting solution with continuous stirring until the solution become homogeneous and clear (Pu et al, 2006). Figure 3-2 shows the steps for preparing the casting solution.

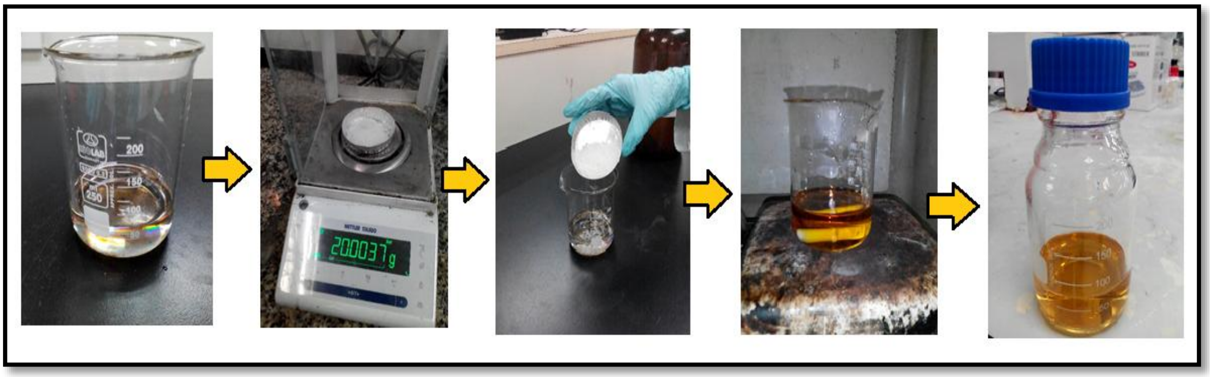

Figure 3-2: Steps for the preparation of casting solution

3.2.2 Casting

The homogeneous solution was then place into a glass plate and cast by using hand-casting knife with a knife gap of 250 mm. The cast was then evaporated for 30 seconds under the surrounding atmosphere. Next, the membrane was immersed into the distilled water. After casting, the membrane was air dried for about one day. The membrane cross-section and surface morphology was determined for every membrane sample by using Scanning Electron Microscope (SEM) (Han et al, 2014). Figure 3-3 shows the steps for casting the membrane.

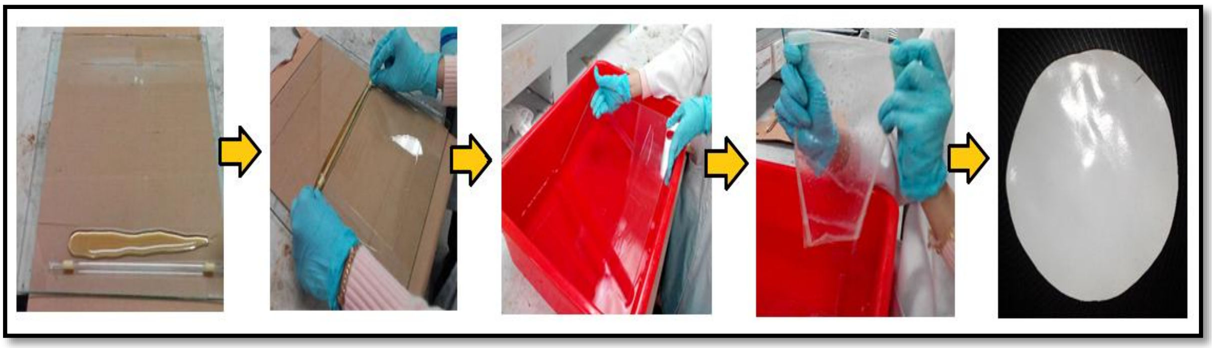

Figure 3-3: Steps for the casting of membrane