Study on the Effects of Dimethylformide in the Preparation of Polycarbonate/Dichloromethane Dope Solution

By

NUR IZNI 'AQILAH ISMAIL

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Universiti Teknologi PETRONAS

Bandar Seri Iskandar

31750 Tronoh

Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

Study on the Effect of Dimethylformide (DMF) in the Preparation of Polycarbonate/Dichloromethane (PC/DCM) Dope Solution

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Nur Izni ‘Aqilah binti Ismail

A project dissertation submitted to the
Chemical Engineering Programme
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Approved by,

____
(Dr. ABRAR INAYAT)

MAY 2014

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original works contained herein have not been undertaken or done by unspecified sources or persons.

(NUR IZNI ‘AQILAH BINTI ISMAIL)

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NUR IZNI 'AQILAH ISMAIL

(CHEMICAL ENGINEERING)

ABSTRACT

Polycarbonate (PC) is well-known in plastic industry, polycarbonate as well used in separation membrane industry. The dissolution of polycarbonate in different aprotic polar solvent will give different result in final cast solution. The reaction when the cast during phase inversion also will show different reaction and various kind of polymer membrane film. It is believed that addition of co-solvent and other variation of preparation condition in the main solvent during the preparation of dope solution will effects much in final membrane film's performance and quality. In this study, the effect of Dimethylformide (DMF) addition as co-solvent in the polymer dope solution is observed in the aspect of density and viscosity. Density of the solution was obtained using manual determination by pycnometer while viscosity is determined using Ubbelohde viscometer. The dope solution used dry phase inversion to form film membrane and the evaporation time is compared by visualization. It was shown that changing the amount of DMF altered the density and the viscosity of dope solution significantly. Other than that, there is limitation where only at certain ratio of co-solvent can be added as co-solvent disturbs the dissolution of polymer.

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CHAPTER 1

INTRODUCTION

1.1 Background study

Membrane simply understood as selective barrier between two phases, having the ability to transport one component to other. Membrane used in broad range of application such as gas separation, sea water desalination, waste water treatment and nitrogen enrichment from air. Different morphology of membrane is required to give off effective separation depends upon the application (M. Iqbal, 2007).

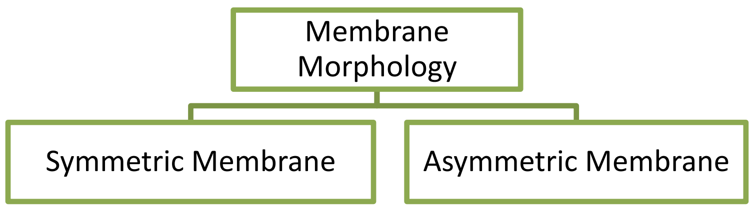


Figure 1: Classification of the typical membrane morphologies (M. Iqbal, 2007)

Polymeric membranes are known for their great flexibility in scale-up, low operating cost and energy efficiency. This has made it widely applied in gas separation. To achieve excellence gas separation performance and continuously enhance polymeric membrane, studies on asymmetric polymeric membrane are often initiated in various aspects or changed parameters. Asymmetric membrane consists of two or more structural planes of non-identical morphologies. Characteristic of asymmetric membrane are specified by its extremely thin and dense film which supported on a thick porous substructure which may be the same or different material of the membrane (Siti Nadiah *et.al*, 2013). Selection of polymer in membrane fabrication is important, dependent on what pair of gas desired to be separated, the preferred porosity or permeability. Often polymers with high T_g (glass temperature) are chosen for gas membrane separation. According to L. M. Robeson (1999), glassy state provides a more structured sieving matrix compared to rubbery state. Other than that, high T_g can bear higher load allowing for high pressure drop across the membrane.

There are few available processes in asymmetric membrane fabrication. Some processes are explained briefly below:

1. Interfacial Composite Membrane

This method coat an aqueous solution onto the surface of micro porous support membrane typically, later immersed in water-immiscible solvent solution containing a reactant. The reactant will react at the interface of the two solutions to form a densely cross-inked, extremely thin layer (M. Iqbal, 2007). Membrane prepared using this method will have a thin dense film of highly cross-linked polymer on the surface if a thicker micro porous support. As the dense polymer is extremely thin on the order of 0.1 μm or less, the permeability is high and high selectivity due to its highly cross-linked.

2, Phase Inversion

Phase inversion process is where transformation of polymer occurs in a controlled way where the liquid polymer solution (polymer plus solvent) changed to solid state. Most dominant of phase inversion is by immersion participation. The polymer solution is first casted on a support plate and immersed in a suitable coagulation bath containing non-solvent. Precipitation occurs due to the exchange of the solvent and non-solvent (M. Mulder, 2000).

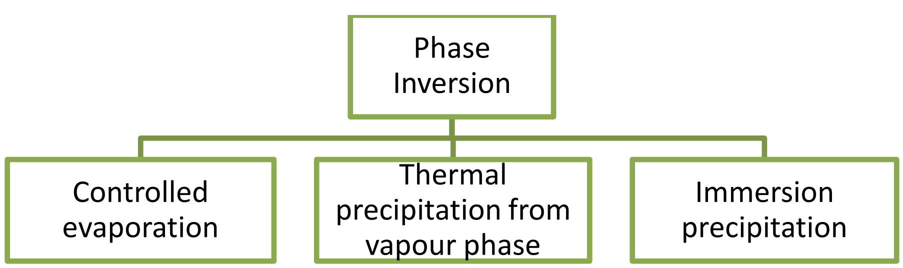


Figure 2: Phase inversion cover ranges of different technique (M. Mulder, 2000)

Structure forming processes during the phase inversion of polymer solution which solvent/non-solvent exchange process occurs are accompanied by various step transformations. L.Yilmaz *et. al* (1986) suggested that mathematical model for this process should consider both kinetic and thermodynamic aspects of the system. To evaluate thermodynamic aspect of the process, ternary diagram seems to be the most suitable tools where complete construction of the ternary phase diagram for the non-solvent/solvent/polymer system will provide information on the formation of a given membrane structure. Addressing kinetic aspect of the system, the modeling involves the construction of proper formulation including mass transfer and concentration profiles during the precipitation process, the moment the casted film immersed in the coagulated bath (L. Yilmaz *et.al*, 1986).

1.2 Problem Statement

Since 1980s, there are various study on non-solvent/solvent/polymer relationship has been initiated. Different type of solvent and parameters are manipulated in order to get a wide range of data and made it beneficial to the enhancement of polymeric membrane. Few techniques have been suggested to improve the performance of the membrane such as adding in additive, vary the dissolution time and temperature, the shear force and many more. Co-solvency is a new spark in membrane fabrication industry where the effect of co-solvent addition, subject to specified ratio and compatibility of co-solvents are still in study. Polycarbonate aromatic asymmetric membrane is one of interesting and potential polymeric membrane, especially in gas separation to give off better results. It is believed that addition of co-solvent can improve the asymmetric matrix of polycarbonate membrane, but a definite combination and performance effect are yet to be known. Therefore, this project research is to focus on the effects of Dimethylformide as co-solvent on membrane performance using thermodynamic study analysis.

1.3 Objective

- **C**onduct a study on the effect of Dimethylformide (DMF) in the preparation of Polycarbonate/Dichloromethane (PC/DCM) dope solution, prior membrane fabrication.
- Focus in different ratio of DMF addition in the main solvent and how the properties of the dope solution affected.

1.4 Scope of Study

In conducting the study, there are several elements that being focused as the scopes of study. The scopes of this study consist of:

- **Composition of solvent use for Polycarbonate dope solution**

  In order to produce asymmetric polycarbonate membrane, people commonly use solvents such as N,N-Dimethylacetamide (DMAc), Dichloromethane (DCM), and N-methyl-2-pyrrolidone (NMP). Each of the solvent has characteristic solubility parameter which determines the polymer dissolution. In this research, Dichloromethane (DCM) has been chosen as main solvent and Dimethylformide (DMF) as the co-solvent. Different composition of the combination was used in the dope solution preparation.

- **Thermodynamic study of the system**

  This research used ternary diagram to compare between pure solution and the one added with co-solvent.

1.5 Relevancy and Feasibility of the Project

By conducting this research, the effect of DMF addition in PC/DCM dope solution will be further study. The finding from this research will be beneficial for the designer and engineers to improve the quality and properties of polymeric asymmetric membrane by referring to the thermodynamic aspect of the system. Equipment and materials needed to perform this research are available in Universiti Teknologi PETRONAS's laboratory thus making this research feasible to be done.

CHAPTER 2

LITERITURE REVIEW

2.1 Polycarbonate

Polycarbonate (PC) was first discovered in 1898 by a German chemist, Einhorn during his attempt to prepare cyclic carbonates by reacting hydroquinone with phosgene. Later in 1902, a similar cross-linked, high molecular polycarbonate encountered by Bischoff and Hedenstrom and the found continued researched by Dr WH Carothers. Finally in the year of 1960, after Bayer and General Electric announced independent development of polycarbonate, they began commercial production [8]. Since then, polycarbonate has been a wide application in many industrial branches as one of main material especially in plastic industries. Compact discs, riot shields, baby feeding bottles, and safety helmets are few of PC typical application.

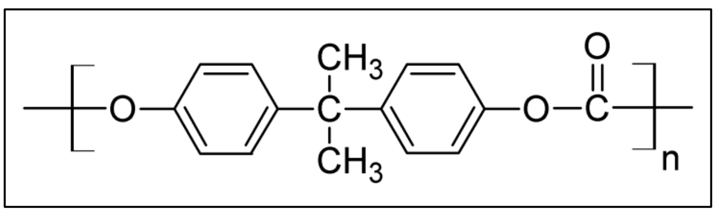


Figure 3: Structure Bisphenol A of Polycarbonate PC

Polycarbonate is famous for its transparency properties, having excellent toughness and good in dimensional as well as thermal stability. PC polymer has two phenyl and two methyl groups in its structure. These two groups contribute much for its molecular stiffness properties resulting in good thermal resistance but relatively high viscosity during processing, due to attraction between phenyl groups and other molecules.

Polycarbonate is also prominent in the study of asymmetric membrane morphology. Morphology properties strongly influence membrane performance and quality. Studies such as the effects on non-solvent additives, polymeric additives, polymer concentration and volatile solvent are few carried out to improve characteristics of separation membrane.

For their high permeability, selectivity and mechanical strength in high pressure application, asymmetric membranes often used for ultrafiltration, gas separation process and reverse osmosis.

2.2 Membrane Fabrication

Phase inversion is one of common method used in fabrication or membrane preparation where a polymer solution loses solvent by evaporation or exchange with another liquid, called non-solvent coagulation, followed by precipitation of the polymer to form the membrane (M. Iqbal *et. al*, 2008).

Membrane preparation will require a dope solution where a polymer is dissolved in specific solvent and given time for dissolution. For the raw material for the dope making, there are few prerequisites to be obeyed (Siemann, 2005):

- The polymer must be soluble in a volatile solvent or water
- A stable solution with a reasonable minimum solid content and viscosity should be formed
- Formation of a homogeneous film and release from the casting support must be possible

The solution will then be cast on glass surface and later, either immersed in non-solvent or allowed to evaporate under ambient or nitrogen environment which further named as dry or wet phase inversion. Comparing these two inversion technique, phase inversion by immersion precipitation is one of the predominant methods used for asymmetric membrane fabrication. Based on the above, we can conclude that there are 3 main components on membrane fabrication;

Figure 4: Three main components on membrane fabrication

2.3 Co-solvency

Solvents that are commonly used to cast of PC membrane are N,N-Dimethylacetamide (DMAc), N,N-Dimethylformamide (DMF), Dichloromethane (DCM), and N-methyl-2-pyrrolidone (NMP). Each of the solvent has characteristic solubility parameter which determines the polymer dissolution. According to Snisarenko *et. al* (2013), to improve the quality of the membrane can be achieved by adding in additives to the dope solution. The additive acts to increase or decrease the solution viscosity, affects the pore formation and the process of phase inversion. In their report, the additives used in the membrane preparation divided into 4; polymeric additive, weak no-solvent, co-solvent and low molecular weight additives.

Co-solvent is a second solvent added into the solution to dissolve the polymer in preparing dope solution. Co-solvent can increase the solubility of a mixture and enhance the chemical stability. Co-solvency or technique of using co-solvent for the stated purposes has been widely used in many industries; for example in liquid drug preparation as well as membrane fabrication (Rubino, 2006). Various ratios and combination of solvents has been investigated to study the effects and improvement to the membrane.

2.4 Thermodynamic Aspect

To understand the mechanism of the membrane phase inversion, thermodynamic and kinetic aspects must be considered. In order to have understanding of the thermodynamic aspect of membrane formation, the ternary phase diagram can be used as a useful source of information. The kinetic aspect should be understood as the rate of solvent-nonsolvent exchange, which is an important factor influencing the structure of pores and morphology of the fabricated membrane.

For thermodynamic aspect, it is often to refer to ternary phase diagram. This diagram provides information about system composition, its behavior in response in addition of any of its components and enables prediction of phase state of the system depending on its composition. The key elements of this diagram are: binodal curve, spinodal curve, a critical point, tie lines, and a glassy (gelation) region. The corners of the triangle represent pure components (polymer, solvent and non-solvent), the axes of the triangle depicts the binary combinations of connected compounds, and any point inside the triangle shows the composition of the system containing all three component.

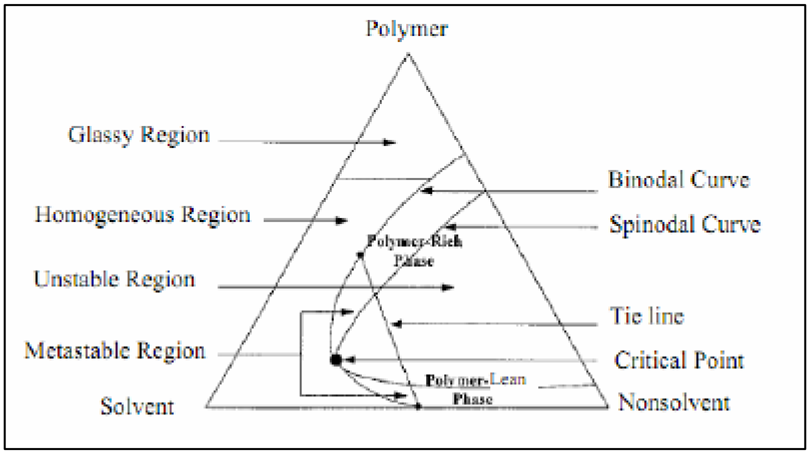


Figure 5: Ternary diagram

Besides thermodynamic aspects, kinetic aspect also should be taken into consideration when studying about membrane formation and effects of co-solvent on morphology. Until today, there are two different approaches has been developed to study the dynamics of phase separation. The first approach is cast-leaching where the coagulation bath composition is monitored. Several works showed that the mass transfer of solvent in coagulation bath from the polymer solution is rate limiting step in coagulation process. In the second approach, the optical microscopy is applied to follow the process of polymer film coagulation.

CHAPTER 3

METHODOLOGY

3.1 Research Methodology

The main purpose of this research is to study on how the addition of DMF affects PC/DCM dope solution. The main methodology used in this researched is based on experiment and discovery as well as study on previous paper work and journals. The research continued with the experiment by modifying basic parameters to various ratios and parameters until the best result obtained. Figure below shows the summary of process routes in conducting this research.

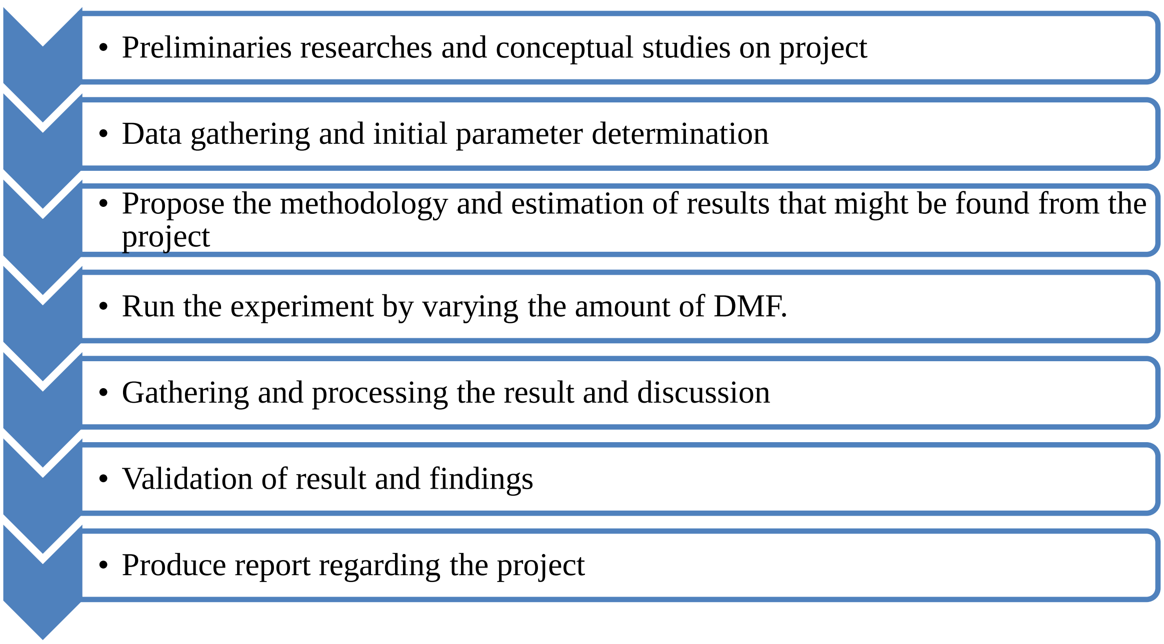


Figure 6: Project research methodology

3.2 Previous Research Related To Effect of Co-Solvent in Polycarbonate Dope Solution

Table 1: Previous related research

Year	Author	Title	Description
2013	Siti Nadiah Mustafa Kamal, Choe Peng Leo, Abdul Latif Ahmad, Mohd Usman Mohd Junaidi	Effects of THF as Co-solvent in the Preparation of Polydimethylsiloxane/Polyethersulfone (PDMS/PES) Membrane for Gas Separation	The study concluded that THF co-solvent resulted in formation of micro and macrovoids in PES membrane. The macrovoid size changing depending on THF concentration in the polymer solution. While, microvoids size increased with the increment of evaporation time prior phase inversion.
2007	Muhammad Iqbal	Development of Asymmetric Polycarbonate (PC) Membrane for Carbon Dioxide Removal from Methane	Effect of various preparation parameters of asymmetric polycarbonate membrane on morphology has been studied, such as solvent/non-solvent pair, evaporation time and non-solvent concentration. The report concluded that, DCM-based membranes have less porous substructure compared to chloroform-based membrane. Other than that, it is shown that, performance of asymmetric PC membrane can be increased by decreasing evaporation time before immersion in coagulation bath.

3.3 Laboratory Experiment

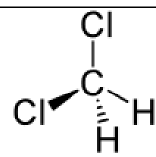
For this project, author has chosen to use below polymer;

Table 2: Properties of Polycarbonate (M. Iqbal, 2007)

	Polycarbonate
Manufacturer	LG-DOW
Type	Amorphous
Characteristic	Good dimensional, shiny surface, high thermal stability, sensitivity to stress cracking
Density (gr/cm3)	1.2
Mr	254 g/mole

The polycarbonate pellet can adsorb moisture from the surrounding environment. Thus, the polymer was dried at 90°C overnight before it is used to prepare the dope solution. For the dope solution, the main solvents are Dichloromethane (DCM).

Table 3: Properties of Solvent (MSDS)

	Dichloromethane (DCM)
	
Molecular Formula	CH_2Cl_2
Molar Mass	84.94 g/mol
Specific Gravity (water=1)	1.33
Appearance	Colorless
%Volatilities	100%

3.3.1 Cloud Point Determination

The determination of cloud point was performed by the titration of polymer solution prepared against Ethanol. The polymer solution was prepared by dissolving Polycarbonate in DCM without and with DMF, sealed and stirred with magnetic stirrer overnight, until the PC is completely dissolved and form a clear transparent homogenous solution. Below composition is used for the dope solution preparation.

Table 4: Cloud point determination solution preparation

DMF Concentration	Amount polymer (15 wt% PC)	Amount DCM (g)	Amount DMF (g)
No DMF added	7.5	42.5	0.00
2	7.5	41.65	0.85
3	7.5	41.23	1.27

The solution is titrated at sealed condition with an air-tight rubber septum stopper through adjustable volume a micropipette. Coagulation was observed in the surface if the solution when droplets of Ethanol dropped into polymer. The solution is continuously stirred until it become homogenous again. Further addition of Ethanol was performed until solution become cloudy and permanently turbid. This point is considered as end point of titration. Ternary phase diagrams of polycarbonate/solvent/coagulant plotted based on the experiments.

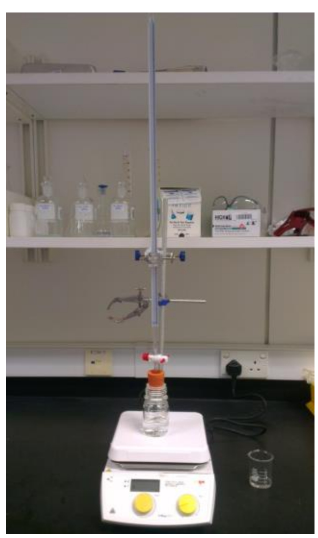


Figure 7: Experiment set-up

Simplest ternary diagram usually created from 3 main components, which are polymer, solvent and non-solvent. As of this project, the polymer is PC; DCM is the solvent and coagulant Ethanol act as the non-solvent component. Ternary diagram is developed using software, Origin Pro version 8.

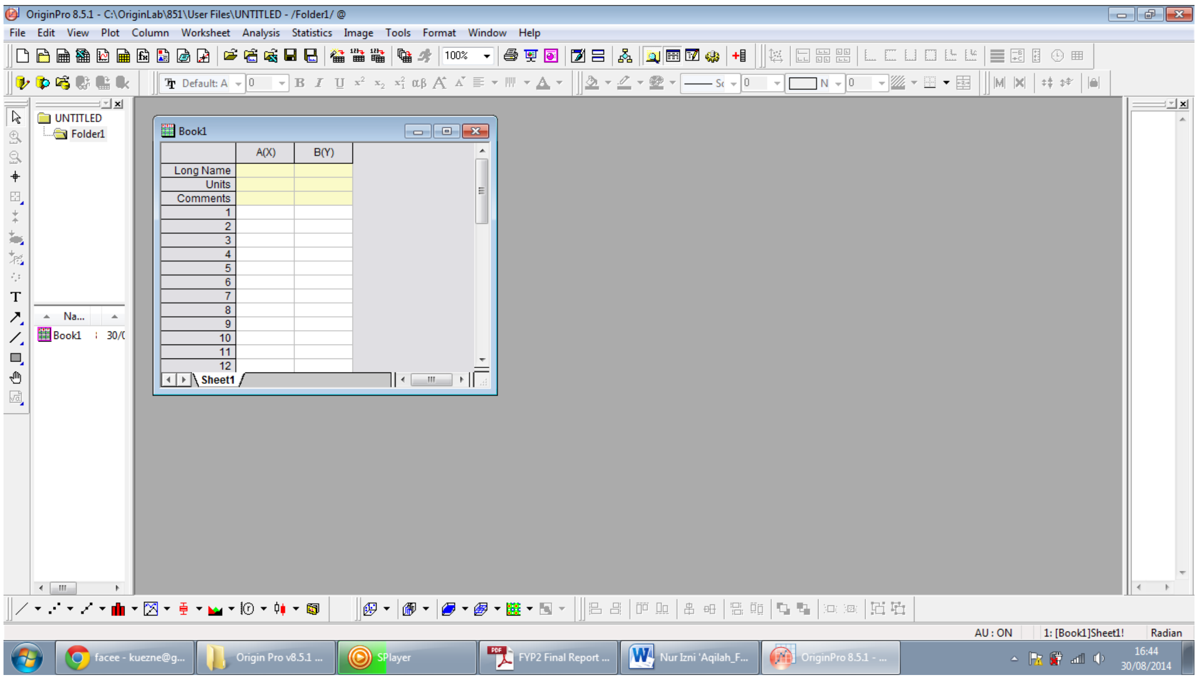


Figure 8: Interface of Pro Origin Software

3.3.2 Solution properties determination

As stated above, the main solvent used in this study is Dichloromethane (DCM) and DMF as the co-solvent. Basic properties studied in this project are density and viscosity. The viscosity is determined using Ubbelohde Ostwald viscometer while the density used manual determination using pycnometer. Ostwald viscometer requires about 20ml of the dope solution. The dope solution is filled in the viscometer, and released from one specified mark to another mark. The time taken for the dope solution to flow down from mark one to the second mark is taken as efflux time. In this project, the viscosity of the dope solution is reported in term of efflux time. We can conclude that the longer the efflux time, the higher the value of viscosity of the dope solution.

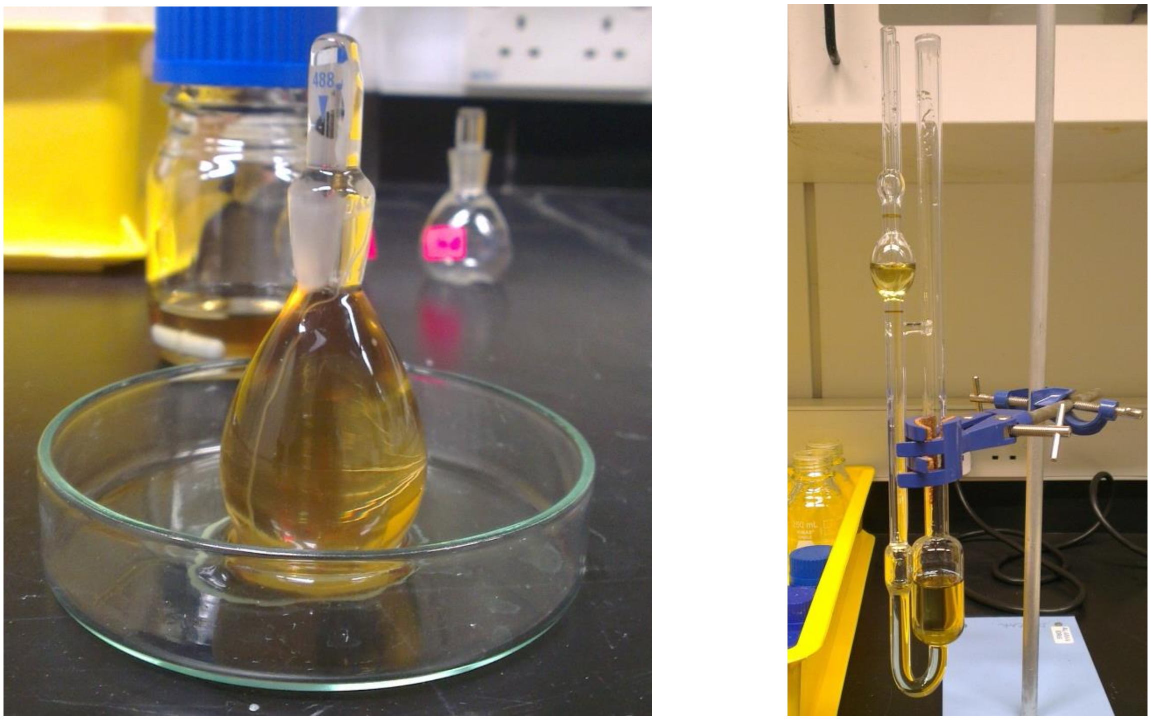


Figure 9: Pycnometer and Ostwald Viscometer experiment set up

For this experiment, the polymer concentration is fixed at 15 wt% of the total solution, a suitable concentration to form film membrane. A total solution of 50 grams was prepared based on weight basis with concentration of 1, 2, 3, 4, 5, 6 and 10 wt% DMF (co-solvent) in polymer solution with DCM as the main solvent.

Table 5: Dope solution preparation

Co-Solvent Concentration	**Amount polymer (15 wt% PC)**	**Amount DCM (g)**	**Amount DMF (g)**
1	7.5	42.08	0.42
2	7.5	41.65	0.85
3	7.5	41.23	1.27
4	7.5	40.8	1.7
5	7.5	40.38	2.12
6	7.5	39.95	2.55
10	7.5	38.25	4.25

The solutions were stirred for 24 hrs with a hot plate magnetic stirrer to achieve homogeneous polymer solutions, in a sealed bottle to prevent any moisture leaked; as DCM vaporizes rapidly.

3.3.3 Wet Phase Inversion

Technically, in membrane separation industry, wet phase inversion membrane fabrication is a more predominant technique used. For wet phase inversion, the dope solution prepared from before was casted on a smooth plane is immersed in a coagulant bath for a certain period of time. For this project, the author chooses to do a dry phase inversion membrane fabrication, and compared the evaporation time taken before the solution becomes a membrane film by visualization. As the study scope should only up to dope preparation only, the wet phase inversion is done to have extra information or data about the effect of DMF addition in PC/DCM dope solution.

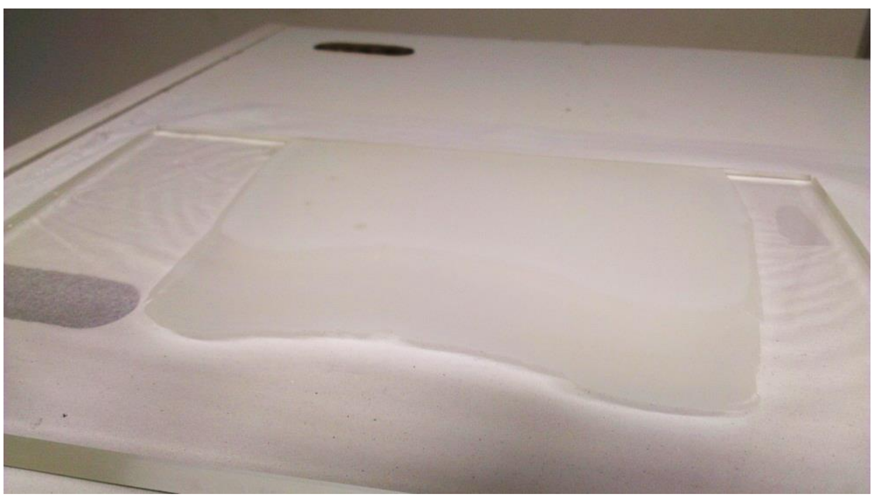


Figure 10: Wet phase inversion membrane fabrication experiment set up

CHAPTER 4

RESULT AND DISCUSSION

4.1 Cloud Point Determination

The coagulation value is defined as the amount of coagulant in grams required to make 100 g polymer solution containing 2 g polymer become turbid (Dongliang, W., et al., 1993) . Coagulant value, basically, indicates the coagulant tolerance of casting solution. Higher coagulation value corresponds to the larger coagulant tolerance of casting solution which causes delayed demixing. The cloud point of ternary system can be conveniently presented using a ternary diagram, which gives more clarity on observing the difference among the different kind of solvents and coagulants used in the experiment.

Ternary diagram was developed using below collected data using Pro Origin software.

Table 6: Cloud point determination data

DMF Concentration	Coagulant needed (ml)
No DMF added	28.0
2	17.5
3	16.5

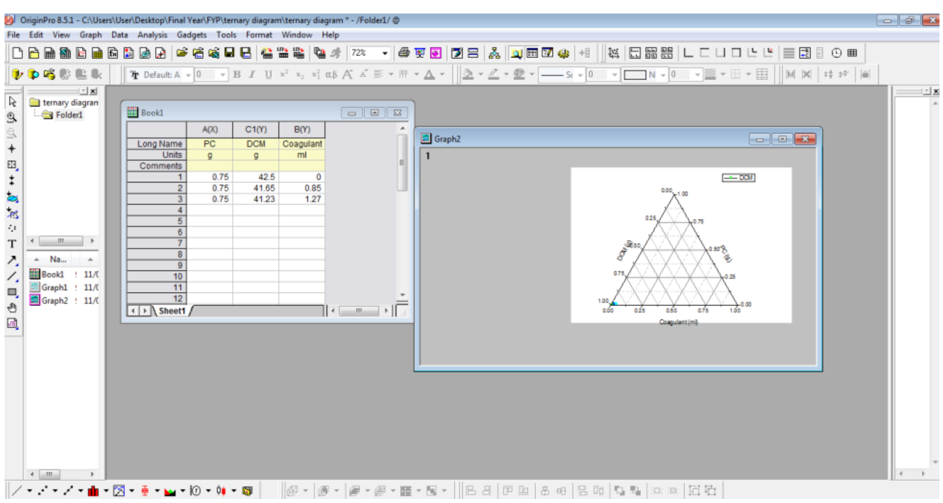


Figure 11: Pro Origin software ternary diagram

Figure 12 shows the coagulation values obtained for pure solution PC/DCM and compared with solution added with DMF at 2% and 3%.

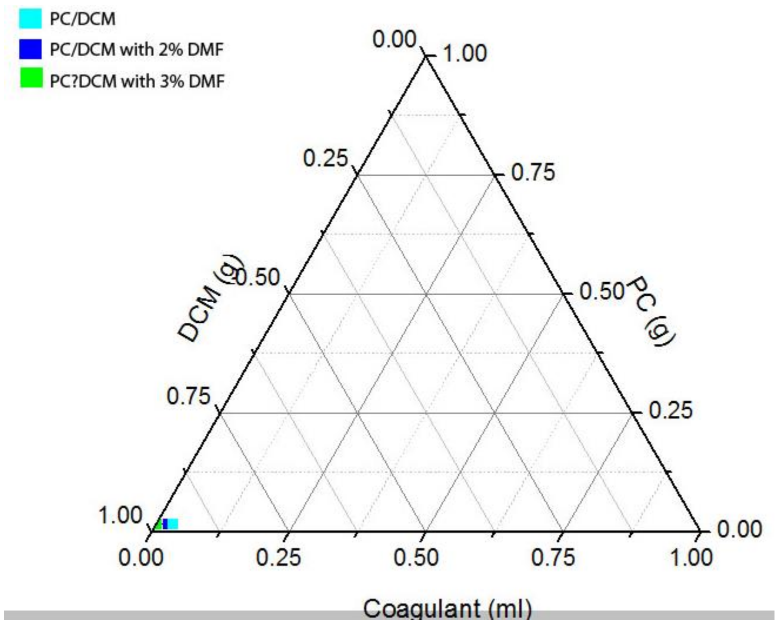


Figure 12: Ternary diagram

Membranes are successfully synthesized when a homogenous polymer solution achieves thermodynamic stability. Co-solvent in polymer dope solution can change liquid-luqid demixing behavior during phase inversion and contributes to changes of membrane morphology and separation properties. From the experiment, solution with higher DMF concentration shifted closer to polymer/solvent axis. This indicates requirement of less coagulant for precipitation to achieve thermodynamic equilibrium compared to the one farther away from the axis. Cloud point closer to polymer/solvent axis exhibit delayed liquid-liquid demixing behavior resulting in membrane with dense layer and closed cell or a spongier substructure.

4.2 Solution properties determination

For this experiment, the polymer concentration is fixed at 15 wt% of the total solution, a suitable concentration to form film membrane. A total solution of 50 grams was prepared based on weight basis with concentration of 1, 2, 3, 4, 5, 6 and 10 wt% DMF (co-solvent) in polymer solution with DCM as the main solvent.

Table 7: Dope solution preparation

Co-Solvent Concentration	Amount polymer (15 wt% PC)	Amount DCM (g)	Amount DMF (g)
1	7.5	42.08	0.42
2	7.5	41.65	0.85
3	7.5	41.23	1.27
4	7.5	40.8	1.7
5	7.5	40.38	2.12
6	7.5	39.95	2.55
10	7.5	38.25	4.25

The solutions were stirred for 24 hrs with a hot plate magnetic stirrer to achieve homogeneous polymer solutions, in a sealed bottle to prevent any moisture leaked; as DCM vaporizes rapidly. The result collected from density and efflux time (viscosity) experiment is tabulated as below;

Table 8: Result for density and efflux time

DMF Concentration	Density (g/cm3)	Efflux time ,s (viscosity)
1	1.3169	1.22
2	1.3132	1.22
3	1.3096	1.23
4	1.3051	1.21
5	1.3005	1.24
6	1.2951	1.24
10	1.2767	161.5

From the data, author can conclude that DMF at small concentration does not give any difference in both properties. At 10 wt%, the viscosity shows significant changes but still the density remains decreasing at a stable rate. As the DMF concentration increases, the dissolving power of solvent will decrease and at the same time making polymer-polymer bonding stronger (M.A Aroon *et. al*., 2010). This leads to incomplete dissolution of polymer or needed a longer dissolution time. Author has prepared solution at higher DMF concentration, the solution indeed need a longer dissolution time up to two or three days for complete dissolution of the polymer. The solution also has become too viscous and impossible to determine the basic properties with manual determination using pycnometer and viscometer.

4.3 Dry Phase Inversion

Visualization of time taken for the dope solution turn into membrane film may not be accurate but a simple hypothesis can be concluded. At higher concentration of DMF, the casted solution form film faster and the membrane is thicker compared to membrane having low DMF concentration. This is compatible to coagulation value result where solution having lower coagulation value has a slower demixing rate and form membrane faster. The membrane of higher DMF concentration also produces a less clear membrane, where porosity of the membrane decreased.

Figure 13: Comparison on membrane film. From left (6 wt%, 5 wt%, 4 wt%, 3 wt% and 2wt% of DMF concentration)

CHAPTER 6

CONCLUSION

Polycarbonate role in separation membrane is becoming dominant from day to day. There are various alternatives has been proposed within the industry on how to increase the performance of the membrane, in order to get a better separation result. Understanding on morphology, separation factor as well as demixing behavior will help in explaining and altering alternatives for separation membrane advanced development.

This project has cover for the basic properties changes after the addition of Dimethylformide into Polycarbonate and main solvent Dichloromethane dope solution. Manual determination of density using pycnometer and viscosity using Ostwald viscometer has been carried out. Other than that, to understand the thermodynamic behavior, the solution is compared by determining its cloud point. Using ternary diagram, it is shown that solution with higher concentration of DMF shifted closer to polymer/solvent axis, where the coagulant value is lower. Theoretically, solution with higher concentration of co-solvent will have delayed liquid-liquid demising rate and a better morphology structure compared to pure solution which has not added any DMF. As this project's scope is prior membrane fabrication, there are suggested recommendations as in Chapter 6 needed to further determine the effects.

The effect of different concentration of DMF in PC/DCM dope solution is studied in the aspect of basic parameter, density and viscosity. From the study, it can be concluded that, only specific amount of DMF can be added into the PC/DCM solution in the means of getting a better performance of the membrane. At optimum polymer concentration 15 wt%, the DMF starts showing effects at 10 wt% addition. Increasing concentration of DMF disturbs the dissolution power of the solvent and results in a less clear/porous membrane film.

CHAPTER 6

RECOMMENDATION

As stated in above chapter, solution at higher DMF concentration has become too viscous and impossible to determine the basic properties with manual determination using pycnometer and viscometer. It is recommended to use other equipment for density and viscosity determination. For viscosity, as the solution is viscous, it is suggested to use fann automated viscometer. This equipment required the solution put in a beaker and rotary part will calculate the viscosity of the solution. The limitation to this equipment are this equipment needed about 50ml to 100ml of solution (which is at bigger quantity to prepared with limited resources) and faster evaporation of the solution during transferring from dissolution beaker to equipment beaker might alter the viscosity value.

Figure 14: fann automated viscometer

Other than that, the fabricated membrane film by dry phase inversion should be proceed to have another **membrane characterization test** to have a more definite and clear analysis on the effect of DMF concentration in the PC/DCM dope solution. Test such as X-Ray diffraction, Gas Permeation and Scanning Electron Microscopy (SEM) test can be done.

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