

DEVELOPMENT OF THERMO-RESPONSIVE  
IONIC LIQUID AS DRAW SOLUTION AND  
ITS PERFORMANCE IN FORWARD OSMOSIS

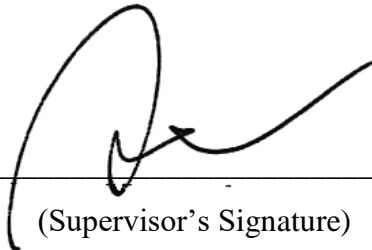
MOHD AMIRUL MUKMIN BIN ABDULLAH

DOCTOR OF PHILOSOPHY

UNIVERSITI MALAYSIA PAHANG

### **SUPERVISOR'S DECLARATION**

We hereby declare that We have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Doctor of Philosophy.



---

(Supervisor's Signature)

Full Name : DR. SYAMSUL BAHARI BIN ABDULLAH

Position : SENIOR LECTURER

Date : 28/12/2020



---

(Co-supervisor's Signature)

Full Name : ASSOC. PROF. DR. SYED MOHD SAUFI BIN TUAN CHIK

Position : ASSOCIATE PROFESSOR

Date : 28/12/2020



## STUDENT'S DECLARATION

I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

A handwritten signature in black ink, appearing to read 'Amirul', is written above a horizontal line.

(Student's Signature)

Full Name : MOHD AMIRUL MUKMIN BIN ABDULLAH

ID Number : PKC 16019

Date : 28/12/2020

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MOHD AMIRUL MUKMIN BIN ABDULLAH

Thesis submitted in fulfillment of the requirements  
for the award of the degree of  
Doctor of Philosophy

Faculty of Chemical and Process Engineering Technology  
UNIVERSITI MALAYSIA PAHANG

DECEMBER 2020

## ACKNOWLEDGEMENTS

In the name of Allah, the Most Gracious and Most Merciful. Glory be to You, we have no knowledge except that which You have taught us. Verily, it is You, the All-Knowing, the All-Wise. Peace and blessings be upon Prophet Muhammad (Peace be Upon Him) and people who walks on the path of His guidance until the judgement day.

I am grateful to Allah for giving me mental and physical strengths to complete this thesis despite obstacles that I have faced during the period of study. I would like to thank several people who have contributed to the completion of this thesis. First and foremost, my deepest appreciation goes to my supervisors Dr. Syamsul Bahari and my co-supervisor Associate Professor Dr. Syed Mohd Saufi for their guidance and encouragement throughout the course of my PhD journey. With their idea and endless support, this research was successfully achieved the target.

My sincere thanks go to all my lab mates and staff members of the Faculty of Chemical and Process Engineering Technology, UMP who helped me in many ways and made my study pleasant and unforgettable. Not to forget, million thanks to Dr. Sharfi, Dr. Mansor, Dr. Fatmawati Latada and postgraduate SERASI team, for their motivation and knowledge sharing throughout this struggle.

I also take this opportunity to thank you to all my friends, especially sahabat MMPM and my housemate Suhaimi, Rasyied Afifi, my neighbors Wong's family, Danish and Darwish and others who directly or indirectly, have lent their helping. In addition, I also indebted to the Ministry of Higher Education of Malaysia under the Fundamental Research Grant Scheme (FRGS/1/2016/TK02/UMP/03/1 - RDU160128) for the research financial support and MyBrain15 scholarship, who provided financial support for my PhD study.

Above all, I would like to extend my deepest gratitude to my beloved mother Haminah Ismail and my late father (Al-Fatehah – till we meet again in Jannah, InsyaAllah), my siblings, nephews, nieces and other family members for their unending support throughout this study. Thank you for the love, encouragement and prayers since the start of this study. I would not make it this far without them. Thank you.

## ABSTRAK

Penyahgaraman berasaskan teknologi membran adalah salah satu pendekatan yang telah banyak diterokai untuk menangani cabaran bagi meningkatkan keperluan air yang bersih. Walaupun proses osmosis berbalik (RO) telah digunakan dahulu, namun teknologi penyahgaraman membran osmosis kehadapan (FO) menjanjikan proses cekap tenaga yang berpotensi serta berdaya maju. Tetapi, masalah utama dalam proses FO adalah kekurangan larutan penarik yang bersesuaian untuk dijana semula. Satu kelebihan yang berbeza menggunakan cecair ionik tindakbalas haba (TRILs) adalah keberkesanan dalam menjana semula larutan penarik melalui rangsangan haba. Dalam kajian ini, 10 kation dan dua anion telah dipilih daripada pangkalan data COSMO-RS dan menerusi kaedah ramalan faktor van't Hoff. 11 jenis ILs telah berjaya disintesis melalui kaedah metatesis dan peneutralan. ILs yang telah disintesis akan dicampur dengan air dan telah disejukkan kepada 0 °C dan kemudian dipanaskan secara beransur-ansur kepada 70 °C untuk menyaring suhu kritikal. Apabila larutan didapati telah terpisah di atas suhu kritikal, campuran ini menjalani fasa peralihan jenis LCST dan yang sebaliknya untuk UCST. Mekanisme interaksi antara molekul TRILs dengan air telah dianalisa menggunakan simulasi COSMO-RS dan H NMR bagi ILs yang terpilih. TRILs telah diuji dalam proses osmosis kehadapan sebagai larutan penarik dan mencapai fluks air yang tinggi, 1-butil-3-metilimidazolium tetrafloroborat ([Bmim] [BF<sub>4</sub>]) (0.71 LMH) dan tetrabutylfosfanium trifloroasetat ([P<sub>4444</sub>][TFA]) (0.44 LMH) berbanding NaCl (0.33 LMH). [Bmim] [BF<sub>4</sub>] dipilih sebagai larutan penarik dimana Aquaporin membran dengan mod PRO telah digunakan untuk menguji kesan parameter dan keadaan optimum untuk proses FO. Eksperimen 2-tahap faktor telah digunakan untuk mengkaji kesan parameter seperti kadar aliran larutan bekalan dan larutan penarik, kepekatan larutan penarik, suhu dan jenis aliran dengan kepekatan larutan bekalan menggunakan air laut tiruan (0.6 M NaCl). Kepekatan larutan penarik dan interaksi antara larutan penarik dan kadar aliran larutan bekalan adalah faktor yang paling penting untuk mencapai fluks air yang tinggi 5.1 LMH. Selain itu, kadar aliran larutan penarik dan interaksi kadar aliran larutan penarik dan bekalan memberi kesan negatif yang ketara untuk mendapatkan fluks garam berbalik rendah 1.3 gMH. Fungsi keboleHINGINAN (DF) telah digunakan, untuk mendapatkan fluks air tertinggi 5.04 LMH dan garam berbalik terendah 1.71 gMH dengan kadar keboleHINGINAN 0.95. Keadaan optimum untuk prestasi FO adalah kadar aliran larutan penarik dan bekalan 300 ml/min dengan kepekatan larutan penarik 3.0 M pada suhu 25 °C dan aliran sehalu. Dalam kajian ini, fasa pemisahan cecair-cecair melalui rangsangan haba boleh dicapai. Jumlah [Bmim][BF<sub>4</sub>] yang dikesan dalam fasa air menggunakan spektroskopi UV-Vis dan air dituliskan menggunakan kaedah penurasan-nano (NF). Tumbuhan kangkung telah dipilih untuk mengkaji kesan kehadiran ILs di dalam air pada pertumbuhan pokok dan jumlah [Bmim][BF<sub>4</sub>] yang dikesan boleh diterima pada kadar kepekatan dibawah 500 ppm. Berdasarkan kepada dapatan kajian ini, dapat disimpulkan bahawa, [Bmim][BF<sub>4</sub>] adalah alternatif yang sesuai untuk digunakan sebagai larutan penarik dalam proses FO.

## ABSTRACT

Desalination based on membrane technology is one of the approaches which has been extensively explored to tackle the challenge in increasing demand of clean water. Although reverse osmosis (RO) process has been applied for a long time, the promising forward osmosis (FO) membrane desalination is viewed as a potentially viable energy efficient performance technology. But, the main problem in FO process is the lack of suitable draw solutes that can be efficiently regenerated. A distinct advantage using thermo-responsive ionic liquids (TRILs) is the efficient in regenerating the draw solute via thermally stimulation. In this study, 10 cations and two anions were selected from COSMO-RS database and van't Hoff factor prediction. 11 type of ILs was successfully synthesized via metathesis and neutralization method. The synthesized ILs were mixed with water and were cooled to 0 °C and then gradually heated to 70 °C to screen critical temperature. When a solution was found to be phase-separated above its critical temperature, this mixture underwent the lower critical solution temperature (LCST) type phase transition and which contrary with upper critical solution temperature (UCST) behavioral. The interactions mechanism of TRILs with water were examined using COSMO-RS simulation and <sup>1</sup>H NMR for selected ILs. The TRILs were tested in FO process as draw solution and achieved high water flux 1-butyl-3-methylimidazolium tetrafluoroborate ([Bmim][BF<sub>4</sub>]) (0.71 LMH) and tetrabutylphosphonium trifluoroacetate ([P<sub>4444</sub>][TFA]) (0.44 LMH) compared to NaCl (0.33 LMH). The [Bmim][BF<sub>4</sub>] was selected as draw solution and aquaporin membrane with pressure retarded osmosis (PRO) mode was used to evaluate the effect of parameter and optimal condition for FO process. The 2-Level factorial experiment design was used to study the effect of parameters such as feed and draw flowrate, draw solution concentration, temperature and type of flow with feed concentration using artificial seawater (0.6 M NaCl). The draw solution concentration and the interactions between draw and feed flowrate was the most significant factors to achieve high water flux 5.1 LMH. Besides that, the draw flowrate and the interaction of draw flowrate and feed flowrate give high significant negative effect which is good to obtain low reverse salt 1.3 gMH. The desirability function (DF) was used, in order to obtain the highest water flux 5.04 LMH and the lowest reverse salt flux 1.71 gMH with 0.95 desirability. The optimal condition for FO performance is 300 ml/min feed and draw flowrate with 3.0 M draw solution at 25 °C and co-current flow. In this research, the phase separation via thermally stimulated liquid-liquid phase separation was achievable. The traceable amount of [Bmim][BF<sub>4</sub>] in water rich phase was detected using UV-Vis spectroscopy and purified using nanofiltration (NF). The water spinach was selected to study the effect of traceable amount of ILs in water on plant growth and acceptable traceable amount of [Bmim][BF<sub>4</sub>] is below 500 ppm. Based on the findings, it can be concluded that, [Bmim][BF<sub>4</sub>] is alternatively suitable to be use as draw solution in FO process.

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