Safety and procedural analysis of membrane separation

Bárbara Martins, Siem Schreurs, *Max Moalin, Maartje Wolters, Evelien Baeten and Kathleen Stout*

Introduction

Studies to create more efficient and sustainable processes are in high demand nowadays, one of those is the implementation of inline separation methods, which can be used to remove the time consumed by work-up steps, such as liquid extractions.
In order to explore this possibility, the potential of membrane technology was studied for two systems: a small scale flow set-up (Zaiput system) and a large scale (500 mL; DSM system), which needed to be first built-up.

Methods

The Zaiput system was tested with two cases:

Case 1: Water and Oil

- 80/20 (%v/v) of water and silicone oil M 5
- Membranes tested: IL-900 and IL-200
- Without and with surfactant (20% Triton X-100)
- Continuous mixing to stabilize
- ^{1}H-NMR analysis

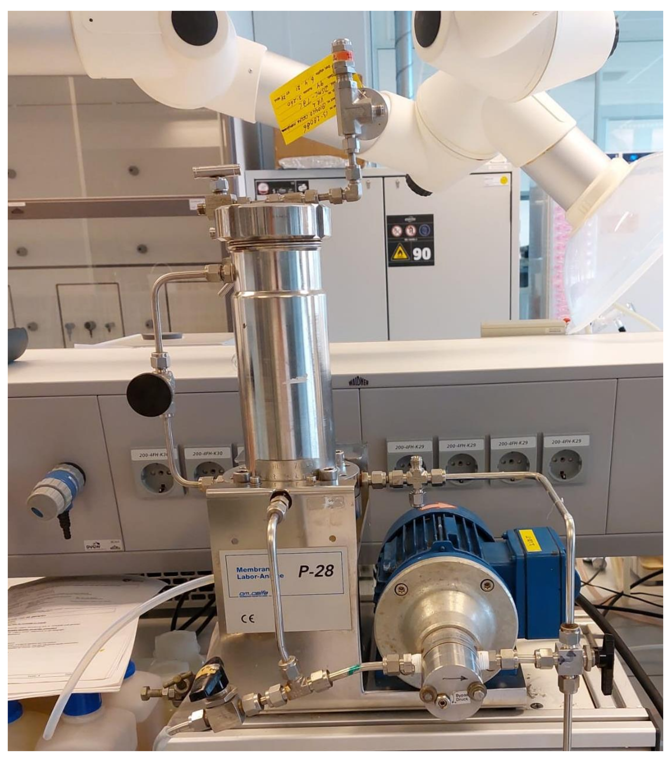
Case 2: Polymer and Monomer

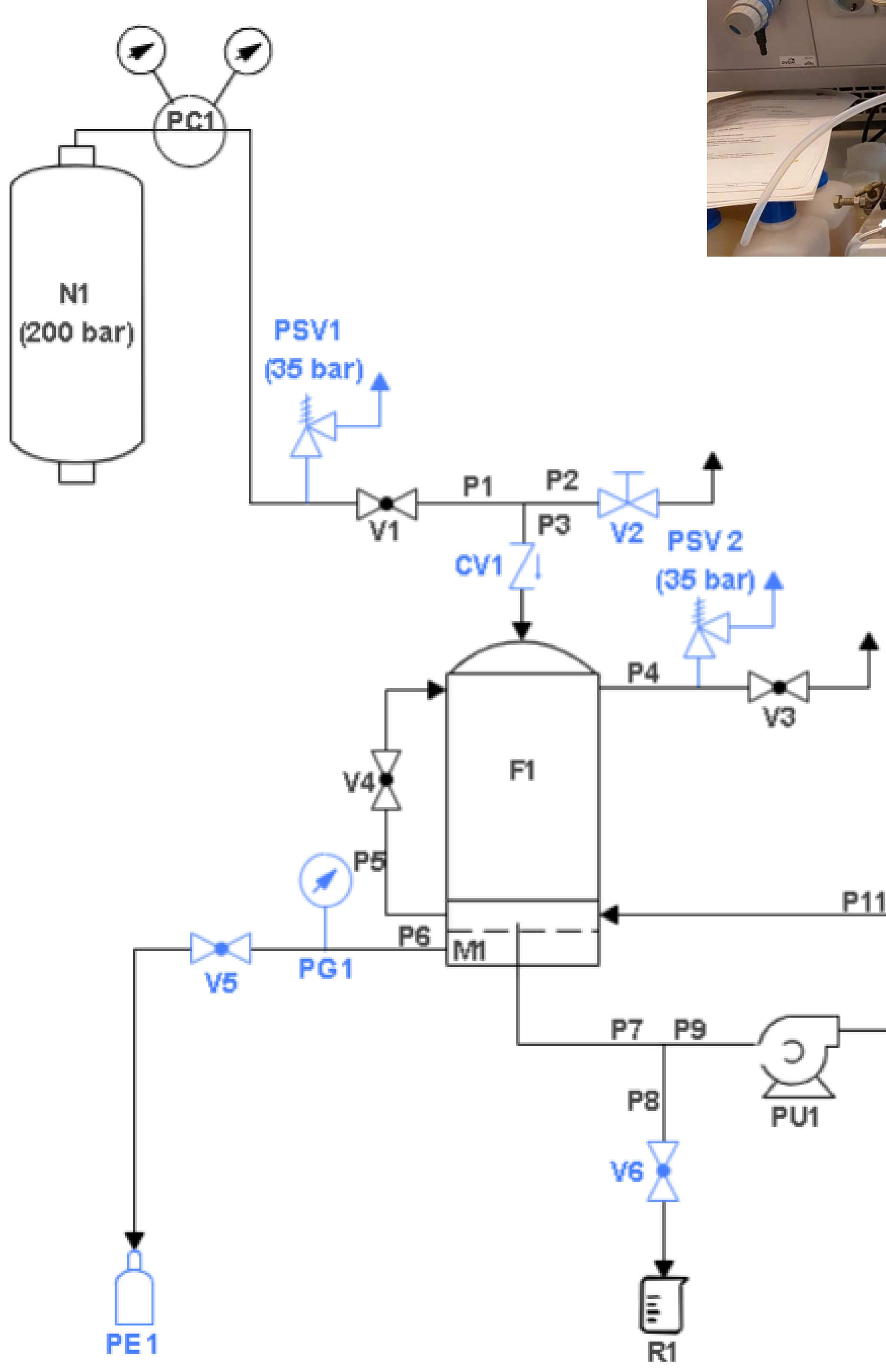
- 2 different polymer sizes: 1,000 and 50,000 g/mol
- Membrane tested: IL-200
- 1:1 monomer (n-BuA) to polymer-monomer unit
- 1:1 (v/v) mixture (polymer + monomer)/n-BuOH
- ^{1}H-NMR and GPC analysis

Results and Discussion

DSM system

- HAZOP (was made to be able to safely use N_2 pressure)
- P&ID
- SOP/Checklist
- Building up the equipment



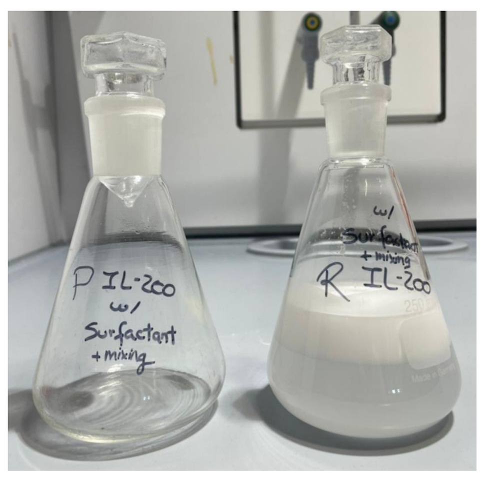


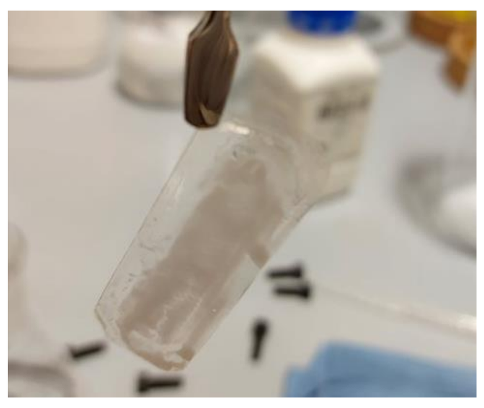
Results and Discussion

Zaiput system

Case 1: Water/silicone oil

	Without Surfactant			
	IL-900		IL-200	
	V_{oil} %	V_{water} %	V_{oil} %	V_{water} %
Feed	24.2%	75.8%	24.2%	75.8%
Permeate	2.5%	97.5%	0.2%	99.8%
Retentate	99.5%	0.5%	99.7%	0.3%

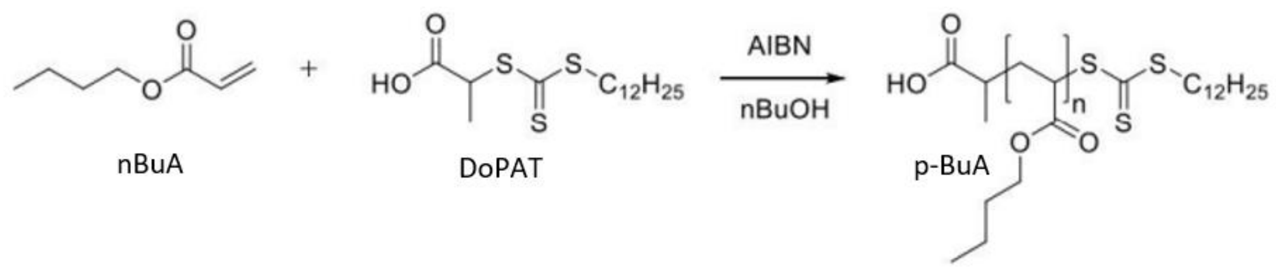


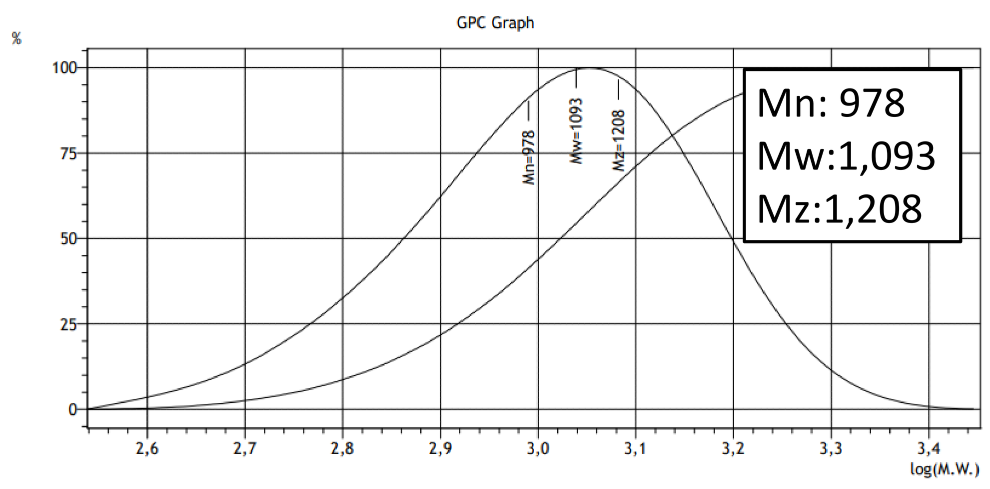


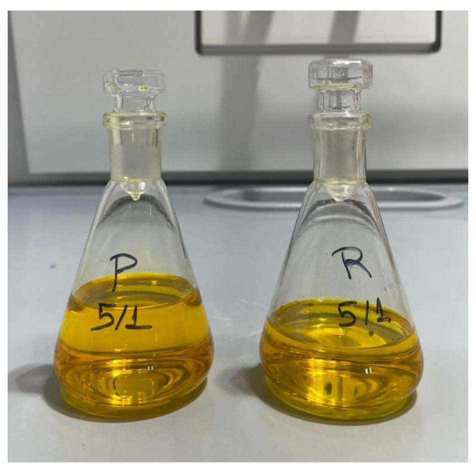
- Without surfactant: best separation with IL-200
- With surfactant: membrane clogged with Triton X-100 and less efficient separation

Case 2: Polymer (p-BuA)/monomer (n-BuA)

- p-BuA was synthetized first







	Polymer 50,000 g/mol	Polymer 1,000 g/mol
	ratio m/p (mmol/mmol)	ratio m/p (mmol/mmol)
Feed	1.37	1.33
Permeate	1.59	0.70
Retentate	1.50	0.70

- No separation for both polymers
- Exit flows were similar: membrane not clogged

Conclusions and Recommendations

- The P&ID and safety assessment have been made for the DSM system
- The separation of oil and water without surfactant was most efficient with the membrane with the smallest pore size
- Upon addition of a surfactant, fouling of the membrane was observed
- No separation or clogging of the membrane was observed during the separation of n-BuA and p-BuA

Recommendations:

- Adaptation of the protocols for the DSM system are needed after the first runs
- The usage of a surfactant with a lower HLB and a membrane with a bigger pore size for the water/oil separation
- The usage of a smaller pore size membrane (from DSM system) and other solvent for the mixture with n-BuOH for the separation of polymer and monomer

Zuyd University of Applied Sciences
ZUYD
UFMG
UNIVERSIDADE FEDERAL DE MINAS GERAIS

Contact: Siem Schreurs - 2064116schreurs@zuyd.nl
Bárbara Martins - 2107536fariasilvamartins@zuyd.nl
Research Centre Material Sciences
Lector Gino van Strijdonck
Faculty of Bèta Sciences and Technology, Heerlen, The Netherlands
www.zuyd.nl/onderzoek/lectoraten/material-science
Chemelot Innovation and Learning Labs, Brightlands Chemelot Campus, The Netherlands

Living Lab Biobased Brazil
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