Research Progress Report

LABORATORY STUDY OF HYDROCARBON FILTRATION

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Content

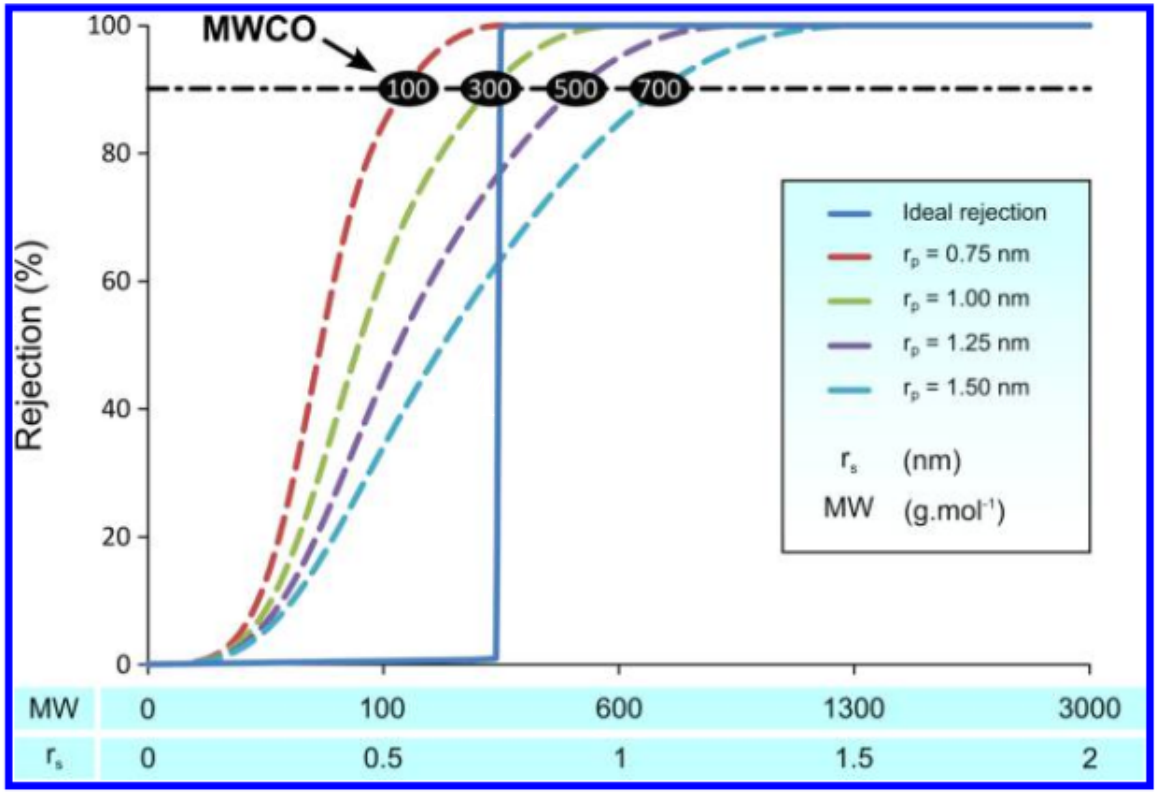
- **Part I:** Literature Review
- **Part II:** Experiment Modification
- **Part III:** Results discussion
- **Part IV:** Conclusions
- **Part V:** Future Work

Part I
Literature Review

Part I: Literature Review

- Organic Solvent Nano-Filtration (OSNF)

Definition: Membrane MWCO/Filtration Efficiency



$$FE = \frac{C_{Pre} - C_{Post}}{C_{Pre}}$$

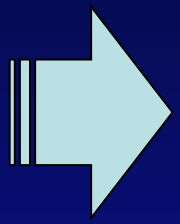
Figure 7. Schematic representation of rejection profile for uncharged solutes by a membrane with uniform pore sizes (r_p) between 0.75 and 1.5 nm by Donnan steric pore-flow model.[36] The dashed lines were obtained by simulation; the solid line represents the desired rejection profile. Adapted with permission from ref 37. Copyright 2013 Elsevier.

P. Marchetti, etc. 2014 Molecular Separation with Organic Solvent Nanofiltration - A Critical Review

Part II
Experiment Modification

Part II: Experiment Modification

- ***Old Membrane:***
- **Puramem Series by Evonik**
  - 280 Da (pore size: ~1.4 nm)
  - Compatible fluid:
    - Aliphatic hydrocarbons (***Hexane***, Heptane)
    - Aromatic hydrocarbons (***Toluene***)
- **PEEK Series by Novamem**
  - PEEK5 (pore size: ~ 5nm)
  - Compatible fluid:
    - Resistance to almost any known organic solvents



- ***New Membrane:***
- **Duramem Series by Evonik**
  - 150 Da to 900 Da (Pore size: ~0.75-4.5nm)
  - Compatible fluid:
    - ***Acetone***
    - Tetrahydrofuran
    - Methanol, Ethanol
    - Methyl-tert-Butyl-Ether
    - Methyl-Ethyl-Ketone
    - Methyl-iso-Butyl-Ketone
    - Butyl Acetate

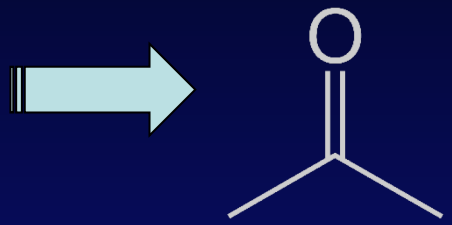
Part II: Experiment Modification

a. Testing Materials [Chemicals]

- **Hydrocarbon Component (Light)**

Hexane (86)

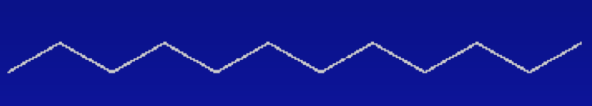
Acetone (58)



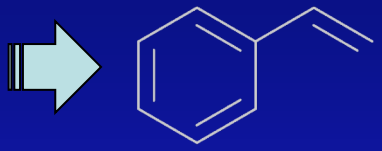
Toluene (92)

- **Hydrocarbon Component (Heavy)**

Dodecane (170)

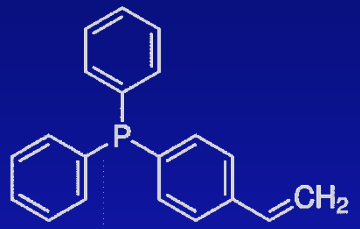


Styrene (104)



MD(236)
α-Methylstyrene Dimer

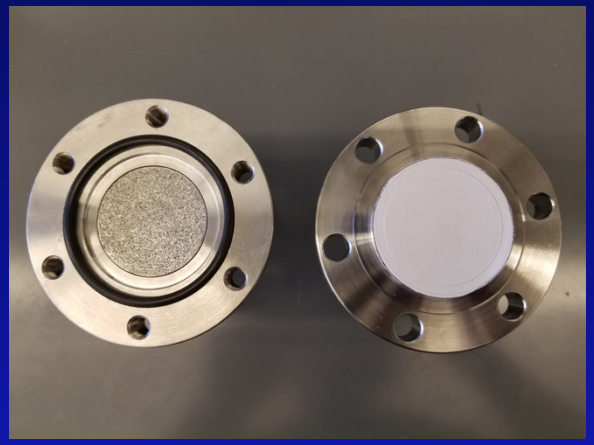
DP-Styrene (288)

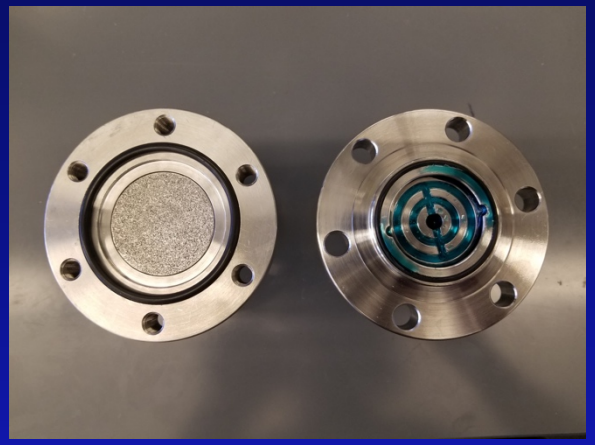


Part II: Experiment Modification

b. Set-Up [Fixing Leakage]

- ***Compatible O-Rings***
  - O-Rings do NOT react with solvents (solutes does not matter)
    - EPR O-Ring: Acetone and Hexane
    - FKM O-Ring: Toluene
- ***Leak Test***
  - Filled the system with blue dyed water under 500 psi; plastic layer in replace of membrane on top with paper underneath





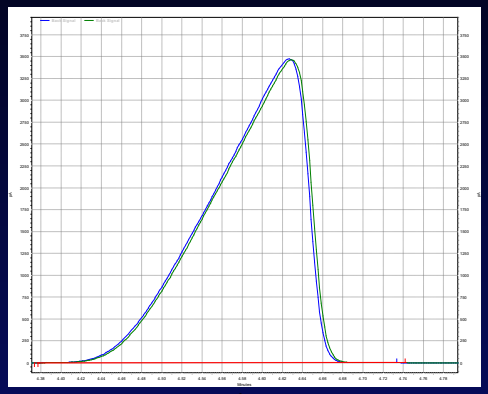
Part II: Experiment Modification

b. Set-Up [Solving Hydrocarbon Precipitation]

- ***Hydrocarbon mixture precipitates when contacting with water***
  - Precipitation clogs tubing, which builds up high pressure (>500 psi)
  - Adapt a new reservoir with piston to separate hydrocarbon mixture and water from pump

Part II: Experiment Modification

b. Set-Up [Schematic]



Membrane Holder

Membrane Inside

Pressure Gauge

Reservoir

Transparent Tubing

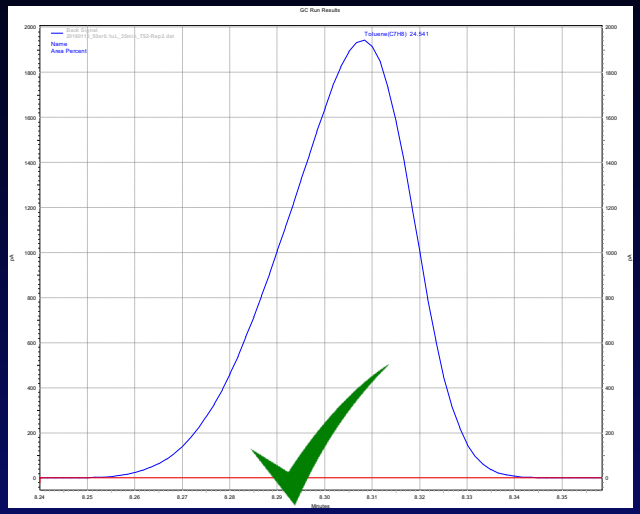
Gas Chromatography

ISCO Syringe Pump

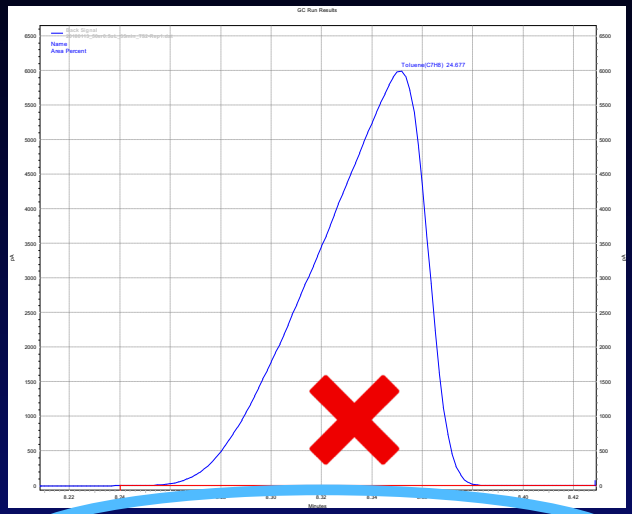
Part II: Experiment Modification

c. Measurement Refinement [GC Parameter Optimization]

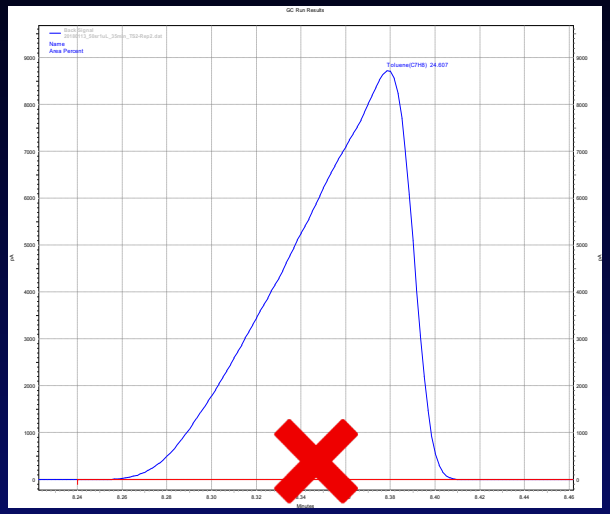
Split Ratio: 50 | Sample Vol: 0.1 uL



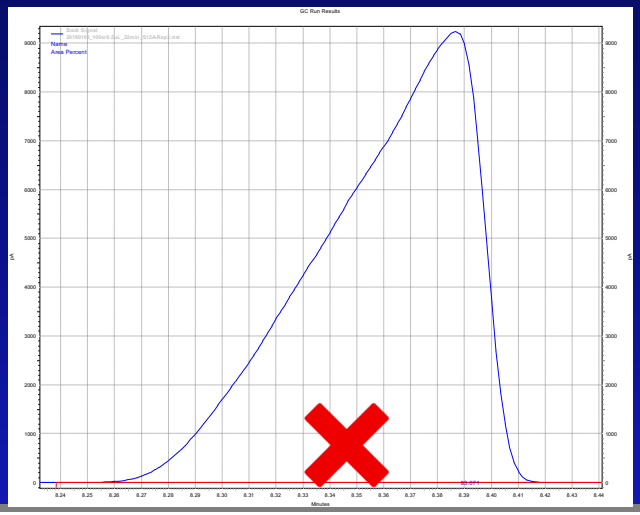
Split Ratio: 50 | Sample Vol: 0.5 uL



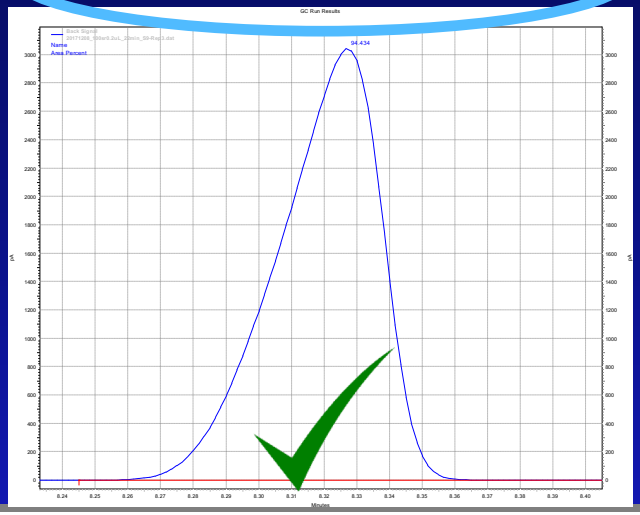
Split Ratio: 50 | Sample Vol: 1 uL



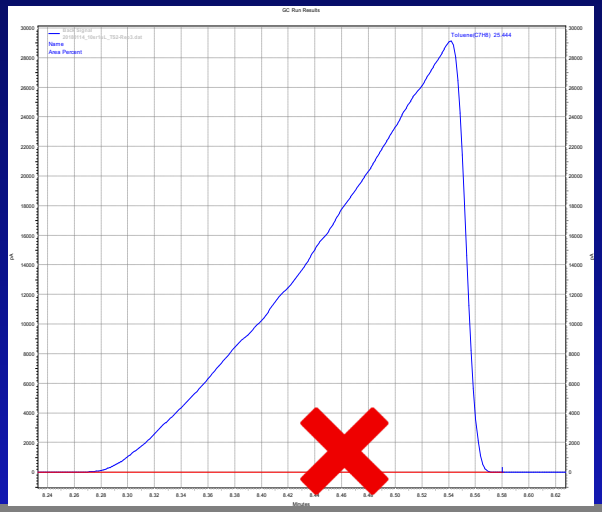
Split Ratio: 100 | Sample Vol: 0.5 uL



Split Ratio: 100 | Sample Vol: 0.2 uL



Split Ratio: 10 | Sample Vol: 1 uL



Part II: Experiment Modification

c. Measurement Refinement [GC Results Reproducibility]

Light Component: Acetone

Before Filtration			
Sample A		ESTD Mole%	Norm Mole%
Rep-1	Light: Acetone	101.07	94.599
	Heavy: Styrene	5.771	5.401
Rep-2	Light: Acetone	101.478	94.591
	Heavy: Styrene	5.803	5.409
Rep-3	Light: Acetone	94.445	94.445
	Heavy: Styrene	5.555	5.555
Rep-4	Light: Acetone	103.491	94.601
	Heavy: Styrene	5.906	5.399
Rep-5	Light: Acetone	108.534	94.607
	Heavy: Styrene	6.187	5.393
Average	Light: Acetone	101.804	94.569
	Heavy: Styrene	5.844	5.431
Std Dev	Light: Acetone	5.0729	0.0693
	Heavy: Styrene	0.2302	0.0693
95% Confidence Interval	Light: Acetone	5.8441	0.0799
	Heavy: Styrene	0.2652	0.0799

Heavy Component: Styrene

Post Filtration			
Sample B		ESTD Mole%	Norm Mole%
Rep-1	Light: Acetone	89.416	94.923
	Heavy: Styrene	4.783	5.077
Rep-2	Light: Acetone	92.602	94.916
	Heavy: Styrene	4.96	5.084
Rep-3	Light: Acetone	92.743	94.912
	Heavy: Styrene	4.972	5.088
Rep-4	Light: Acetone	87.721	94.912
	Heavy: Styrene	4.702	5.088
Rep-5	Light: Acetone	89.764	94.901
	Heavy: Styrene	4.823	5.099
Average	Light: Acetone	90.449	94.913
	Heavy: Styrene	4.848	5.087
Std Dev	Light: Acetone	2.1723	0.0080
	Heavy: Styrene	0.1163	0.0080
95% Confidence Interval	Light: Acetone	2.5026	0.0092
	Heavy: Styrene	0.1340	0.0092

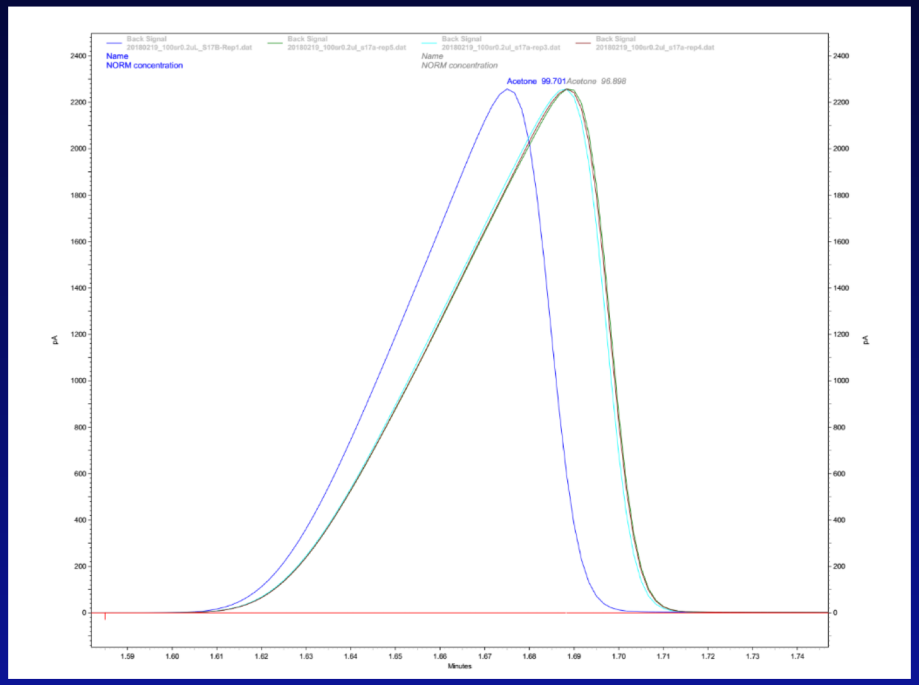
Improvement: standard deviation reduces by half from ~0.15% to ~0.08% !

Part III
Results Discussion

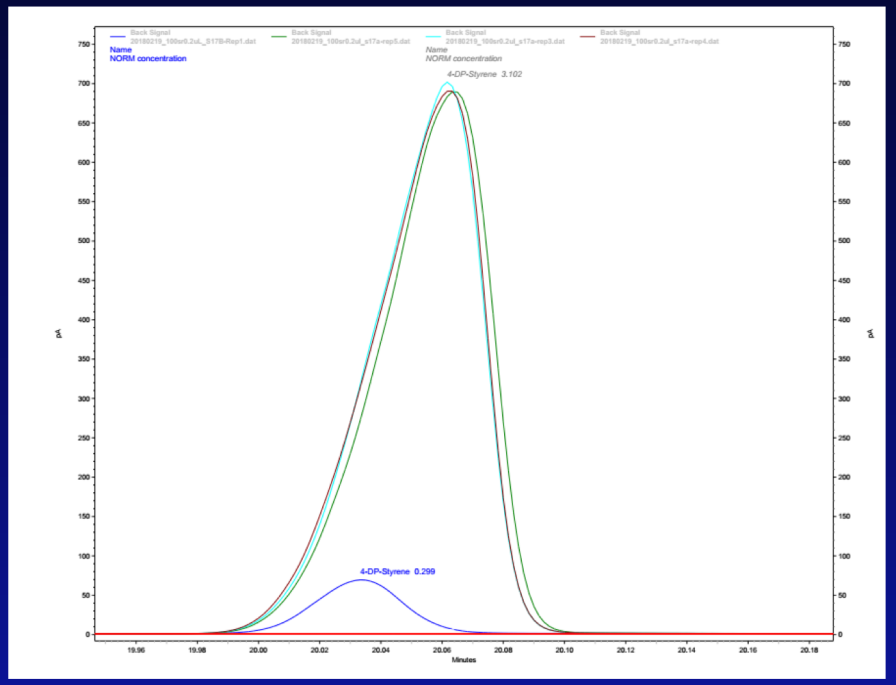
Part III: Results Discussion

a. Example of GC Results [Before and Post Filtration]

Light Component: Acetone (Normalized)



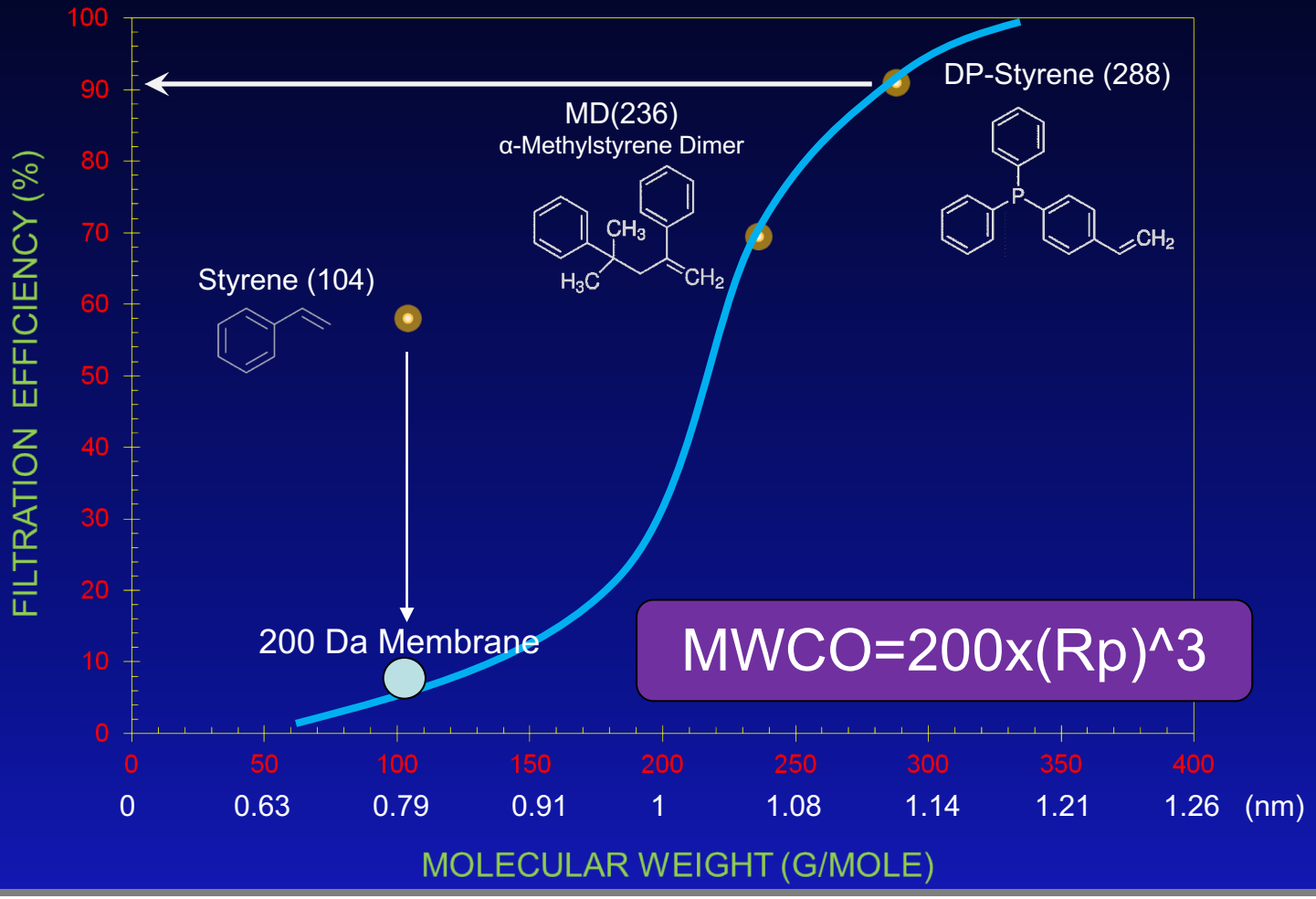
Heavy Component: DP-Styrene



Part III: Results Discussion

b. Filtration Efficiency vs. Molecular Weight

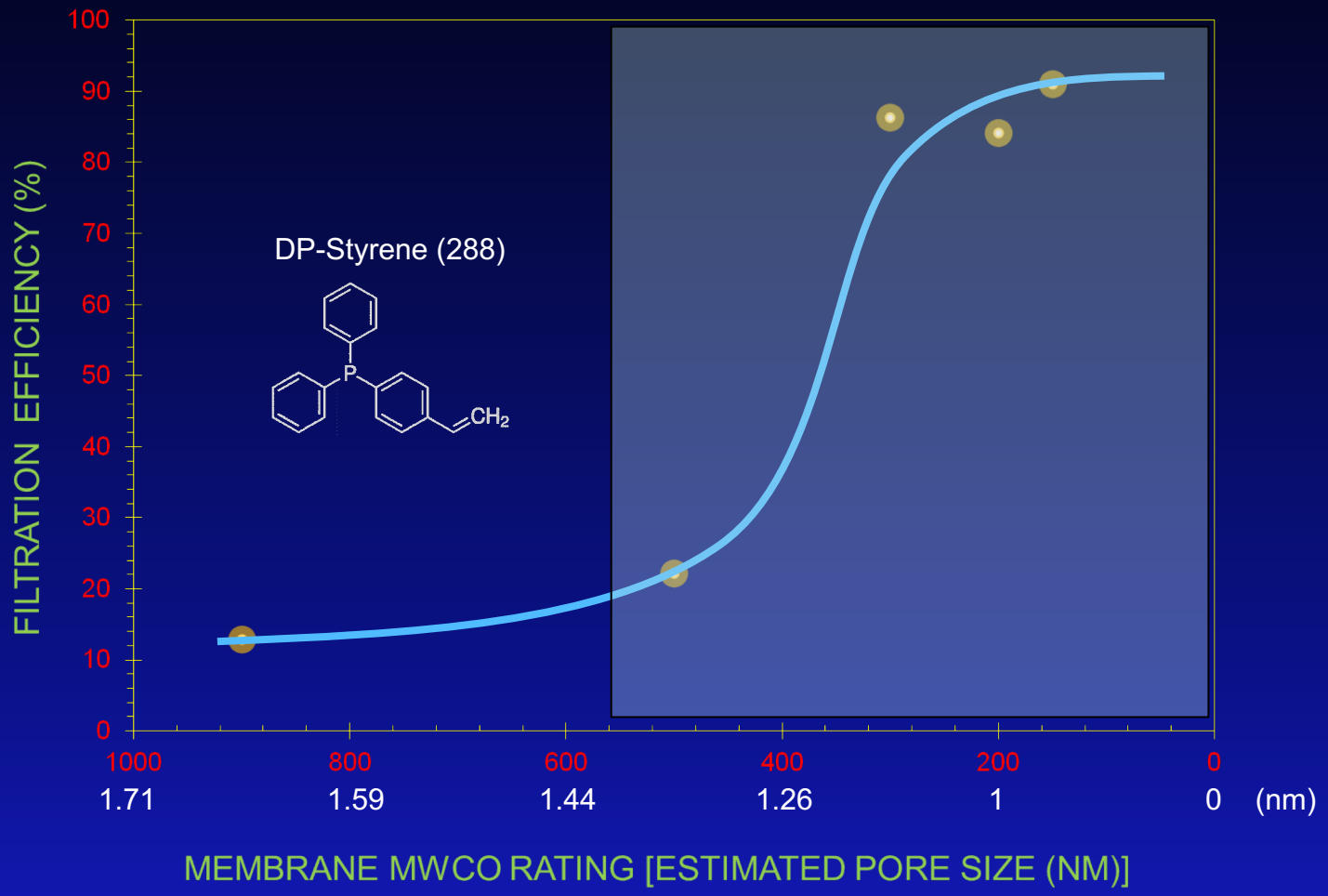
Duramem 150 Da under ~200 psi



Part III: Results Discussion

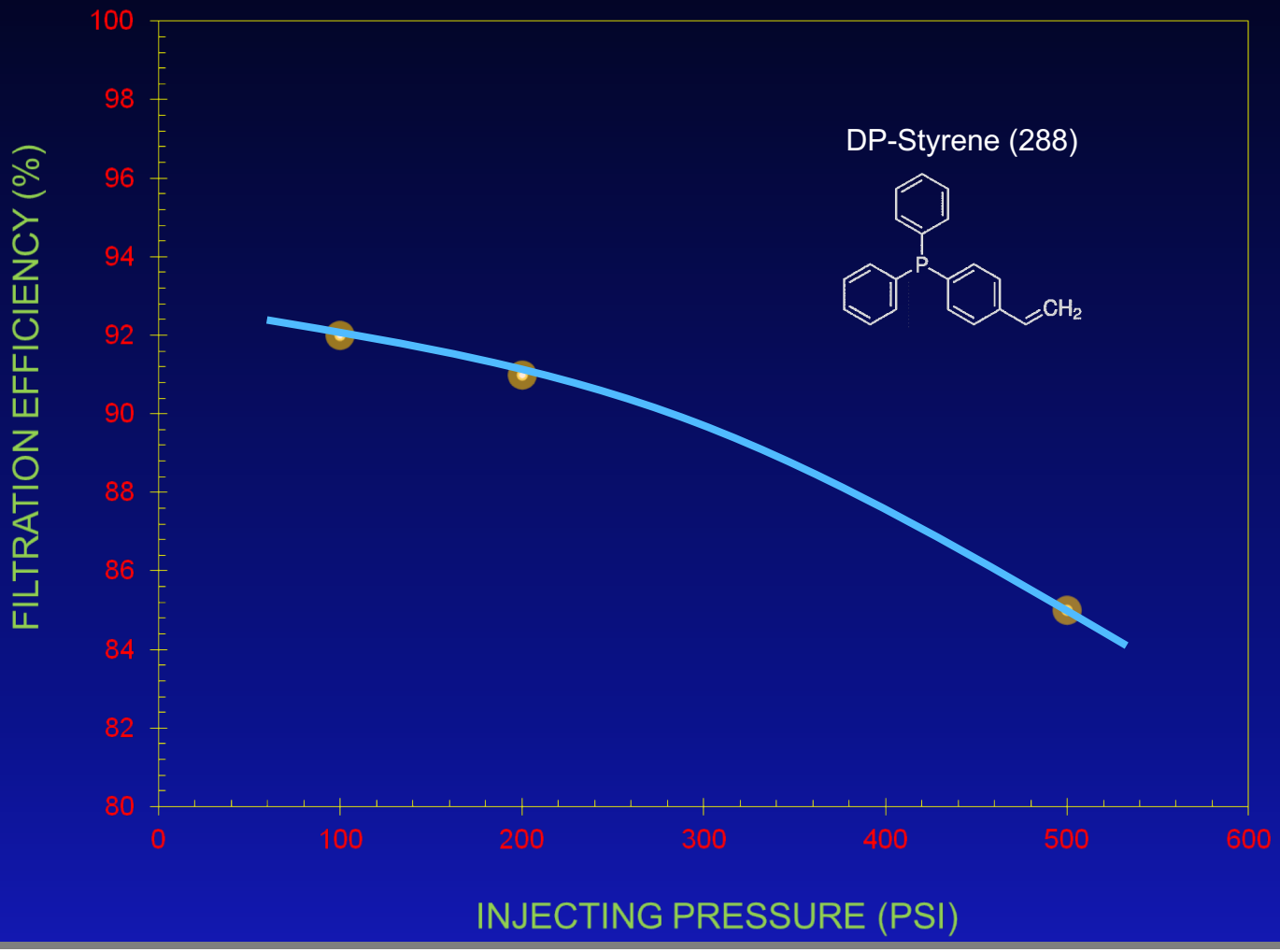
c. Filtration Efficiency vs. Membrane Pore Size

Various Duramem with DP-Styrene (288 Da) under ~200 psi



Part III: Results Discussion

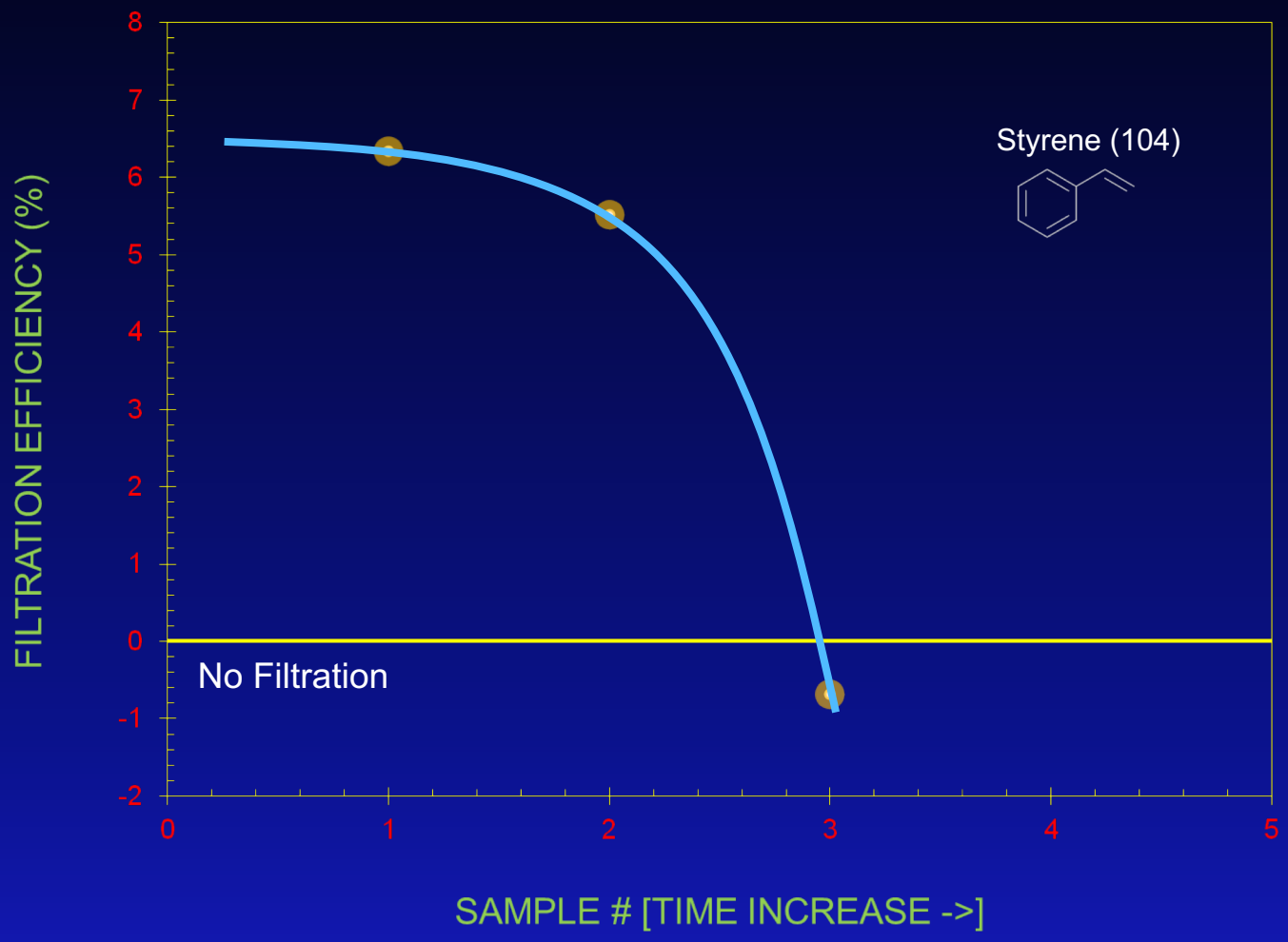
d. Filtration Efficiency vs. Injecting Pressure



Part III: Results Discussion

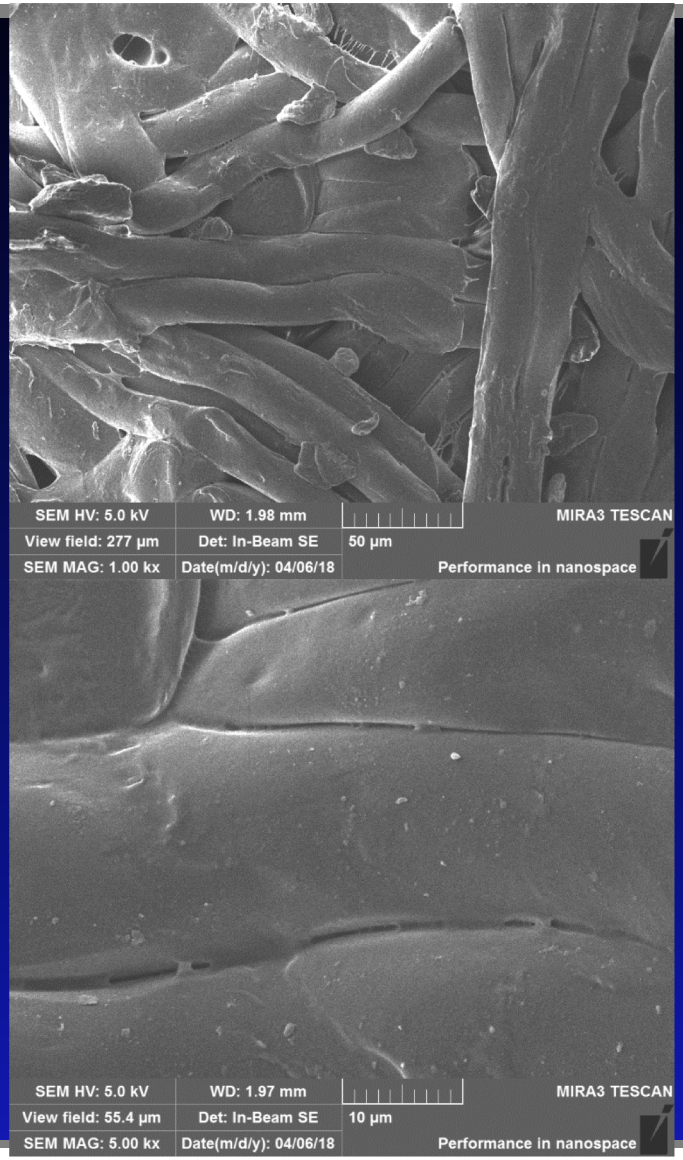
e. Filtration Efficiency vs. Time

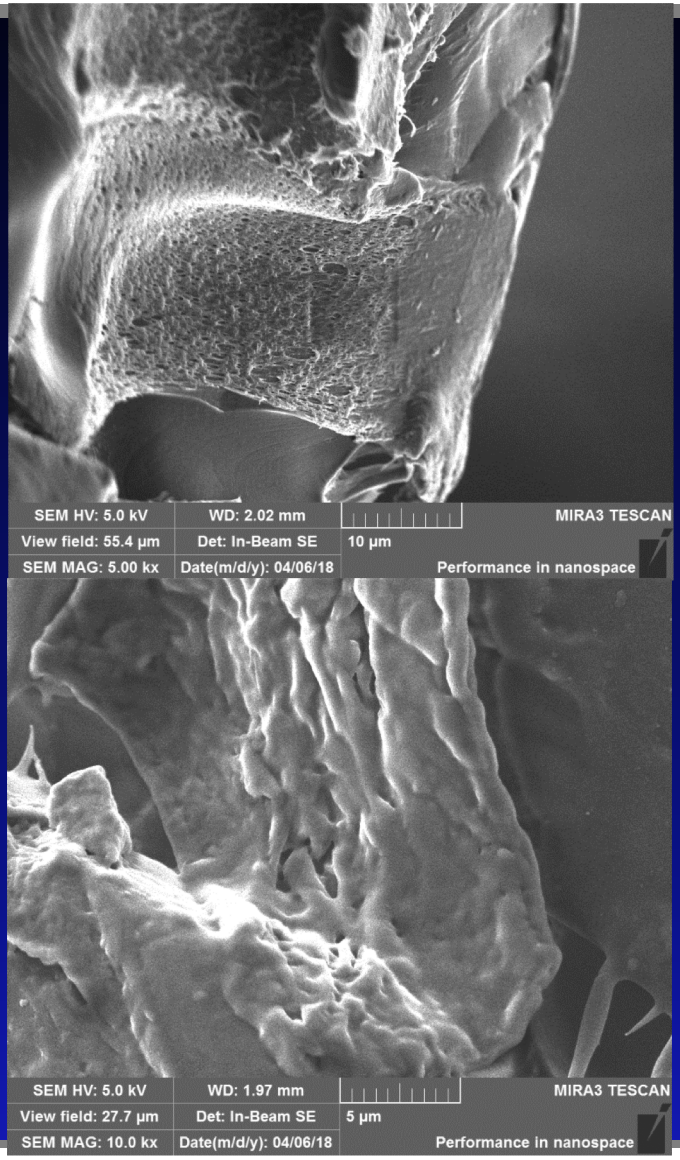
Duramem 200 Da with Styrene (104 Da)



Part III: Results Discussion

f. SEM Image of Membranes [Duramem 150 Da Supporting Side]

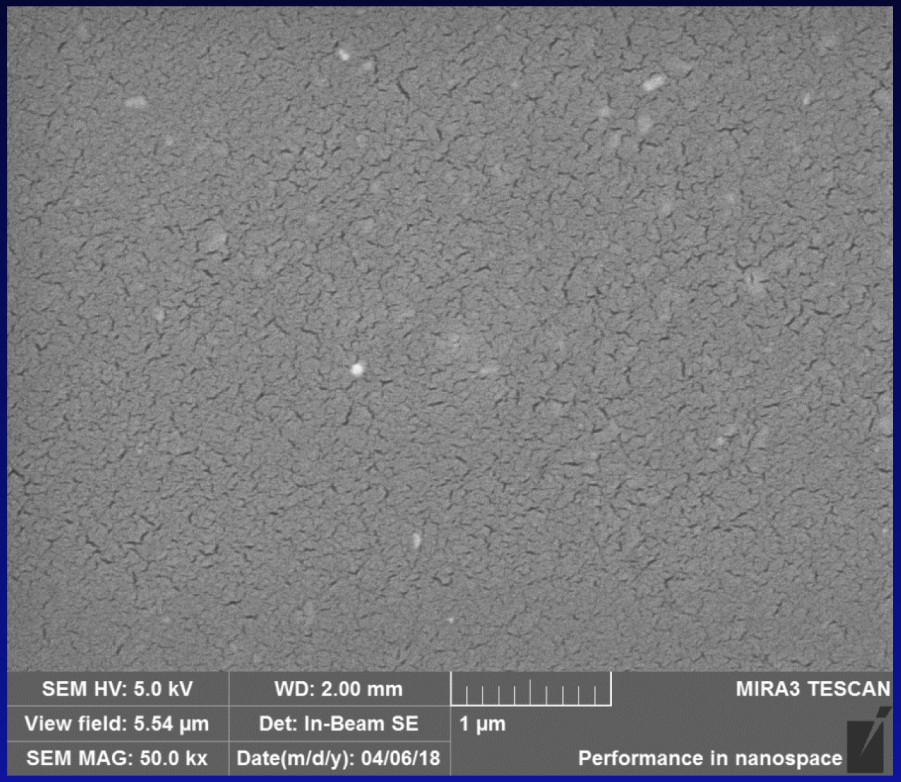




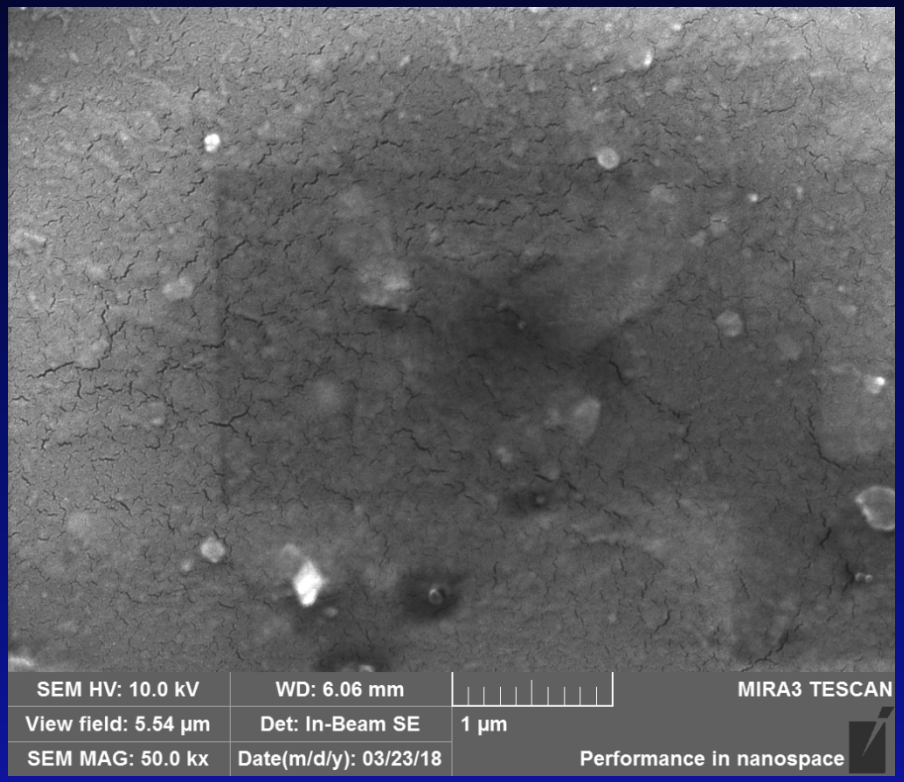
Part III: Results Discussion

f. SEM Image of Membranes [Duramem 150 Da Working Side]

Brand New



Used



Part IV
Conclusions

Part IV: Conclusions

- ***Filtration of hydrocarbon through artificial membranes has been verified in the lab***
  - Filtration efficiency (or rejection percentage) is proportional to hydrocarbon molecular weight and inverse proportional to membrane pore size (or MWCO rating)
  - Tests showed filtration of hydrocarbon whose molecular weight is lower than MWCO of membrane, i.e. the kinetic diameter of molecules is smaller than the membrane pore size
    - The filtration efficiency decreases with time
  - Filtration efficiency decreases with increase pressure

Part V

Future Work

Part V: Future Work

- *Set-Up Modification*
  - Design a device to collect effluent samples easily at different time
- *Additional Measurement Data*
  - Effluent pressure measurement
- *New Chemicals*
  - Branched vs linear chain heavy hydrocarbons
- *Experimental Condition*
  - Change of temperature
- *More testing of … … ?*

Questions and Suggestions?