Research Summary

EXPERIMENTAL STUDY OF HYDROCARBON FILTRATION

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Content

INTRODUCTION

Background

- Motivation
  - Compositional differences have been observed between hydrocarbons produced on surface and hydrocarbons in the reservoir
  - The micron to nanometer scale of shale pore size leads us to suspect the filtration of hydrocarbon by shale reservoir during the production
- Strategy
  - Verify filtration effect through shale 'core flood' (Z.Zhu)
  - Study filtration mechanism through artificial membrane

    - Simplified and controlled experimental parameters

Examples of Shale Pore Size Distribution

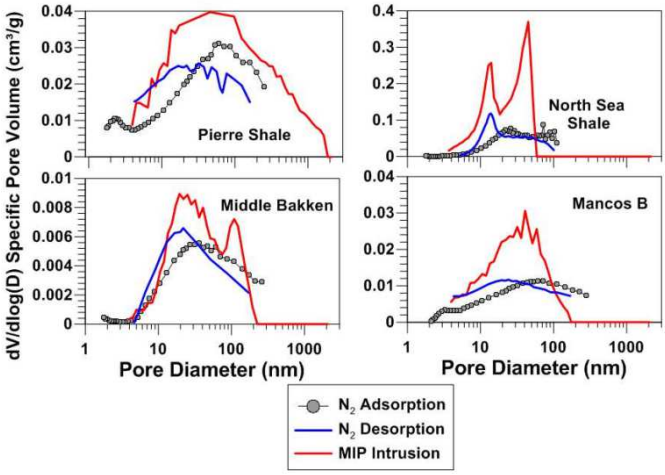


Figure 4.21: Comparison of PSD obtained from N_2 adsorption (gray, N_2 desorption (blue) and MIP intrusion (red) branch from four mudrock formations.

K. Kuila, PhD thesis, Measurement and Interpretation of Porosity and Pore Size Distribution in Mudrocks: The Hole Story of Shales

Filtration Spectrum

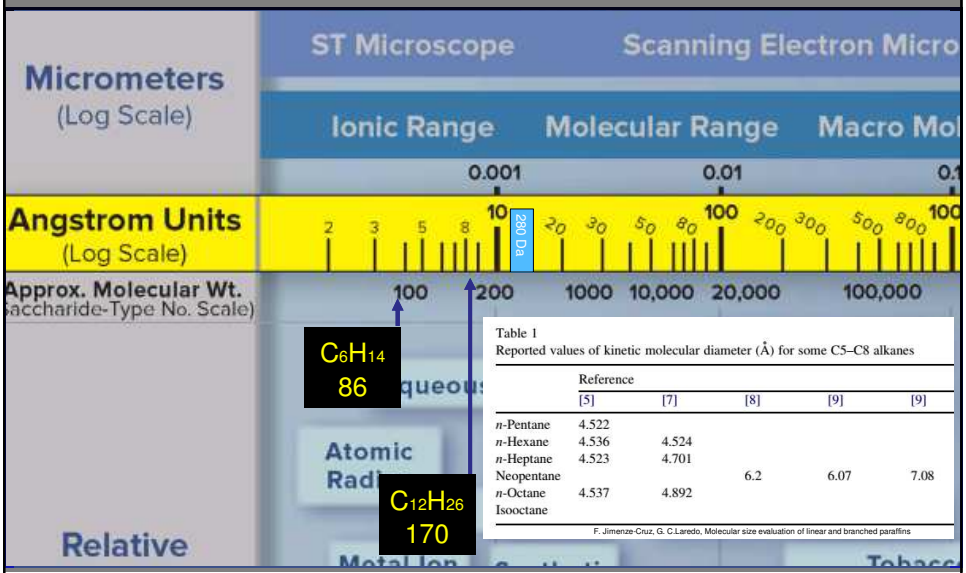


Table 1
Reported values of kinetic molecular diameter (Å) for some C5–C8 alkanes

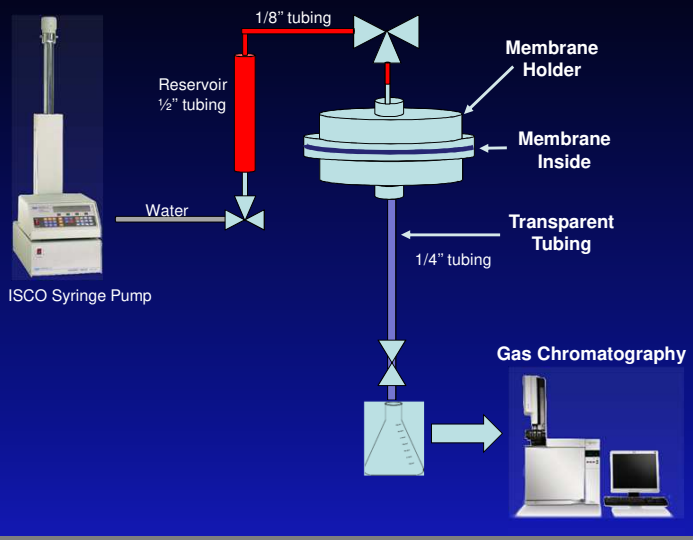
	Reference				
	[5]	[7]	[8]	[9]	[9]
n-Pentane	4.522				
n-Hexane	4.536	4.524			
n-Heptane	4.523	4.701			
Neopentane			6.2	6.07	7.08
n-Octane	4.537	4.892			
Isooctane					

F. Jimenze-Cruz, G. C.Laredo, Molecular size evaluation of linear and branched paraffins

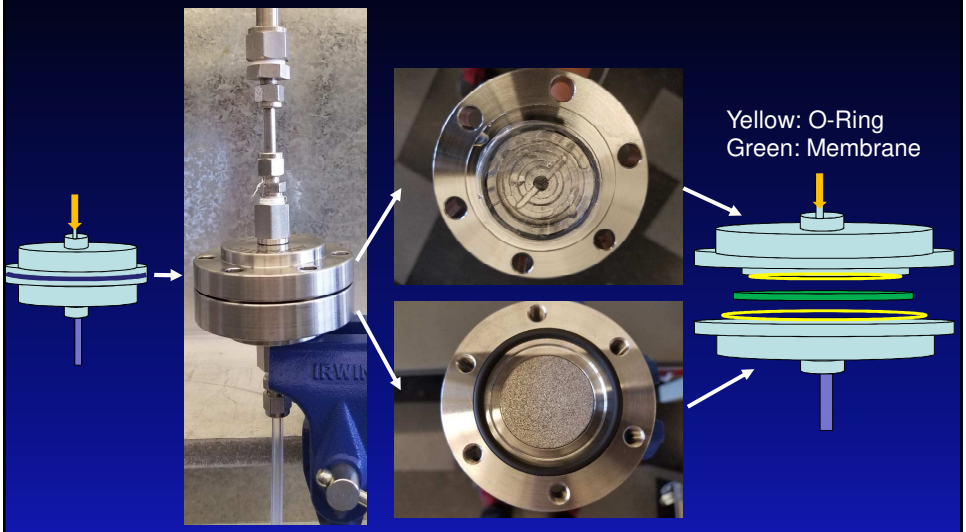
*Filtration spectrum from Sterlitech

EXPERIMENTAL SET-UP

Schematic of Experimental Set-Up



Experimental Set-Up

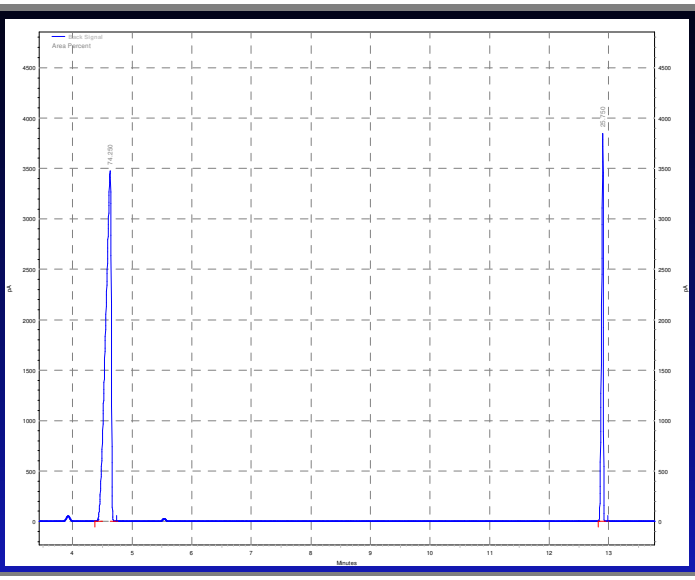


Set-Up Integrity Test

- Leak test
  - There was no leak when system was pressurized to 300 psi (constant pressure controlled through pump) for 24 hours
- Test S5: 4 layers of 280 Da membrane under **300 psi**
  - Flow **upward**
  - **Found leak** of fluid outside of holder
- Test S6: 2 layers of 280 Da membrane under **200 psi**
  - Flow **upward**
  - **Found leak** of fluid outside of holder
- Test S7: 1 layer of 280 Da membrane under **25 psi**
  - Flow **downward**
  - **No leak** found
- ***Conclusion: Fluid arriving at downstream tubing or leaking outside of holder has been flowed through membrane***

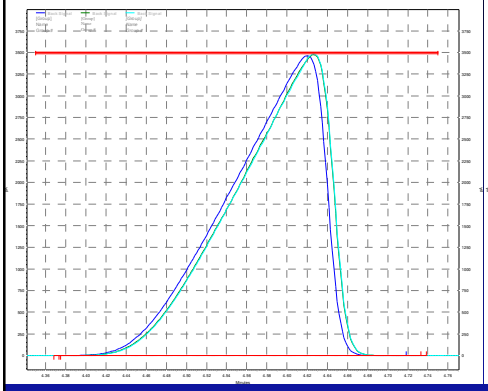
GAS CHROMATOGRAPHY RESULTS

Gas Chromatography Example (S6A-2)

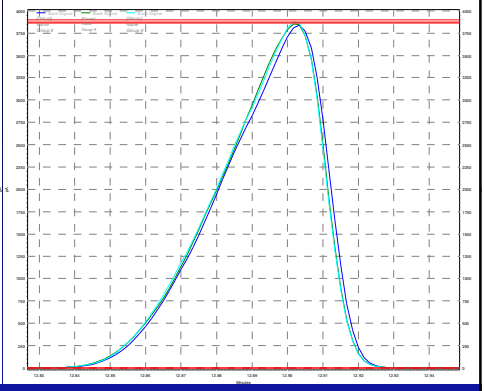


Gas Chromatography Example (S6A and S6B)

Before filtration:S6A, 3 reps

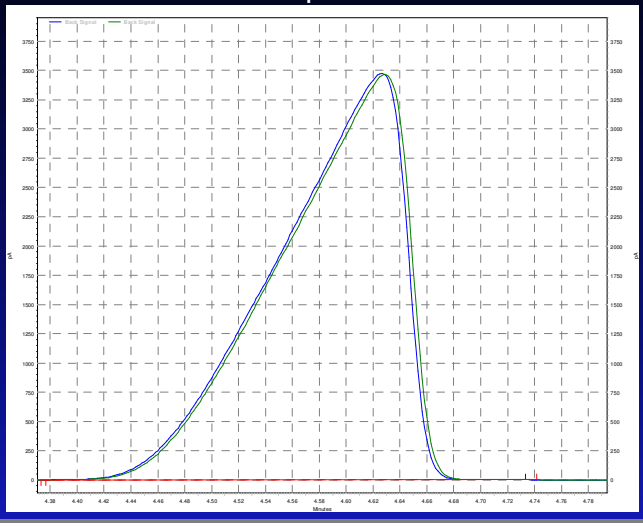


After filtration: S6B,3 reps



Gas Chromatography Example (S6A-2 and S6B-2)

Before and filtration comparison:S6A-2 and S6B-2



Test S5: 4 Layers 280 Da Membranes, 300 psi, Upward

Sample #	GC Run #	C6 (%)	C12 (%)	Sum(%)
S5A	S5A-1	74.902%	25.098%	100.000%
	S5A-2	74.681%	25.319%	100.000%
	S5A-3	74.628%	25.372%	100.000%
Largest Variance		0.221%	0.221%	
Standard Deviation		0.145%	0.145%	

Sample #	GC Run #	C6 (%)	C12 (%)	Sum(%)
S5B	S5B-1	74.029%	25.971%	100.000%
	S5B-2	73.975%	26.025%	100.000%
	S5B-3	74.074%	25.926%	100.000%
Largest Variance		0.045%	0.045%	
Standard Deviation		0.050%	0.050%	

Sample #	GC Run #	C6 (%)	C12 (%)	Sum(%)
S5	S5A-2	74.681%	25.319%	100.000%
	S5B-2	73.975%	26.025%	100.000%
S6B-S6A		-0.706%	0.706%	
Standard Deviation		0.499%	0.499%	

Test S6: 2 Layers 280 Da Membranes, 200 psi, Upward

Sample #	GC Run #	C6 (%)	C12 (%)	Sum(%)
S6A	S6A-1	74.191%	25.809%	100.000%
	S6A-2	74.250%	25.750%	100.000%
	S6A-3	74.196%	25.804%	100.000%
Largest Variance		0.059%	0.059%	
Standard Deviation		0.033%	0.033%	

Sample #	GC Run #	C6 (%)	C12 (%)	Sum(%)
S6B	S6B-1	74.059%	25.941%	100.000%
	S6B-2	74.093%	25.907%	100.000%
	S6B-3	74.066%	25.934%	100.000%
Largest Variance		0.007%	0.007%	
Standard Deviation		0.018%	0.018%	

Sample #	GC Run #	C6 (%)	C12 (%)	Sum(%)
S6	S6A-2	74.250%	25.750%	100.000%
	S6B-2	74.093%	25.907%	100.000%
S6B-S6A		-0.157%	0.157%	
Standard Deviation		0.111%	0.111%	

Test S7: 1 Layer 280 Da Membrane, 25 psi, Downward

Sample #	GC Run #	C6 (%)	C12 (%)	Sum(%)
S7A	S7A-1	74.025%	25.975%	100.000%
	S7A-2	73.946%	26.054%	100.000%
	S7A-3	74.023%	25.977%	100.000%
Largest Variance		0.079%	0.079%	
Standard Deviation		0.045%	0.045%	

Sample #	GC Run #	C6 (%)	C12 (%)	Sum(%)
S7B	S7B-1	74.025%	25.975%	100.000%
	S7B-2	73.625%	26.375%	100.000%
	S7B-3	73.580%	26.420%	100.000%
Largest Variance		0.445%	0.445%	
Standard Deviation		0.245%	0.245%	

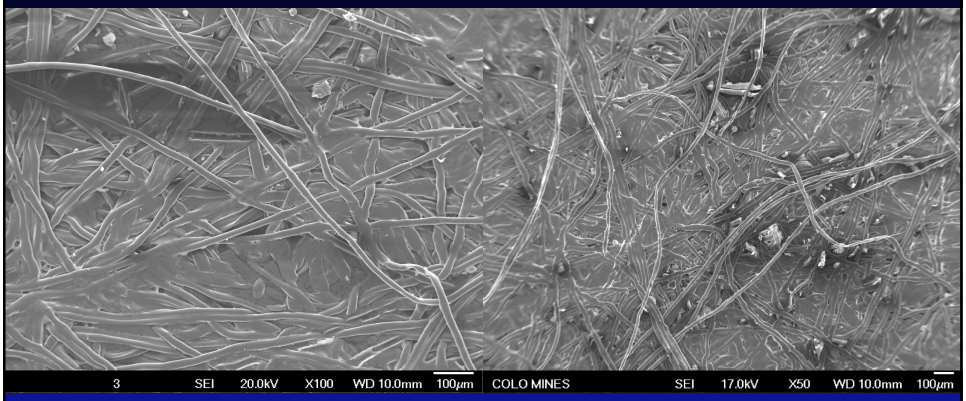
Sample #	GC Run #	C6 (%)	C12 (%)	Sum(%)
S7C	S7C-1	73.669%	26.321%	99.990%
	S7C-2	73.679%	26.331%	100.010%
	S7C-3	73.676%	26.324%	100.000%
Largest Variance		0.010%	0.010%	
Standard Deviation		0.005%	0.005%	

Sample #	GC Run #	C6 (%)	C12 (%)	Sum(%)
S7	S7A-2	73.946%	26.054%	100.000%
	S7B-2	73.625%	26.375%	100.000%
	S7C-2	73.679%	26.331%	100.010%
S7B-S7A		-0.321%	0.321%	
S7C-S7A		-0.267%	0.277%	
Standard Deviation		0.172%	0.174%	

SEM IMAGES OF MEMBRANES

SEM of Membranes (S5:4 layers 280Da, 300 psi)

Before Filtration (100x) | After Filtration (100x)



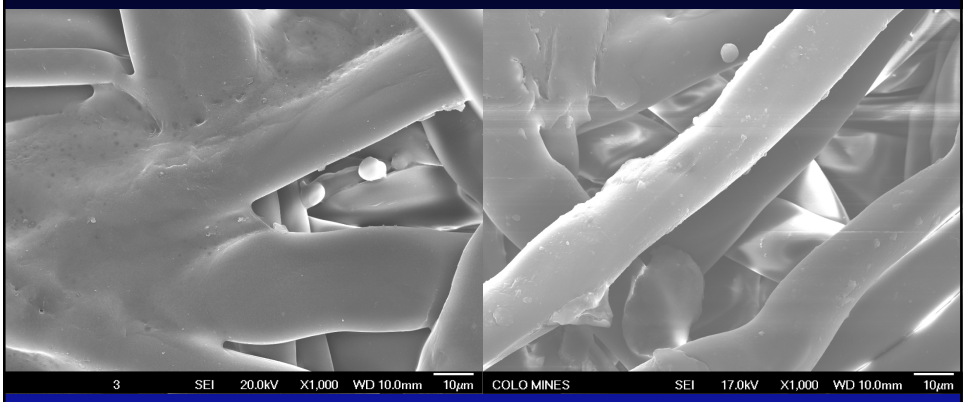
300

SEM of Membranes (S5:4 layers 280Da, 300 psi)

Before Filtration (500x) | After Filtration (200x)

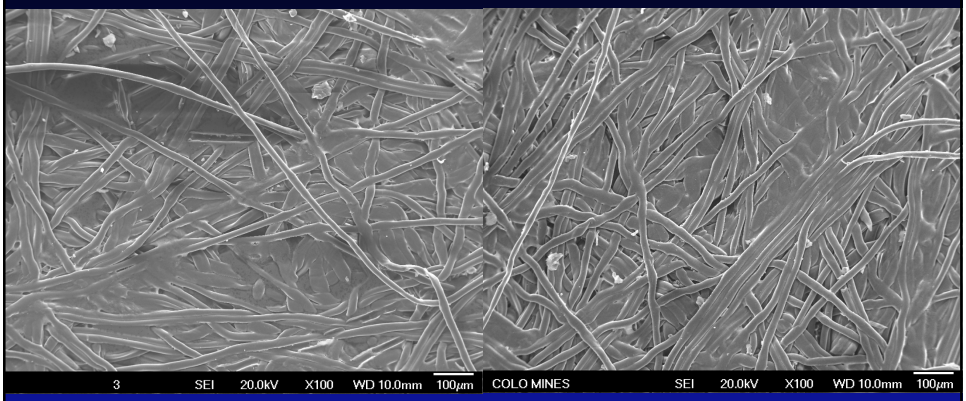
SEM of Membranes (S5:4 layers 280Da, 300psi)

Before Filtration (1000x) | After Filtration (1000x)



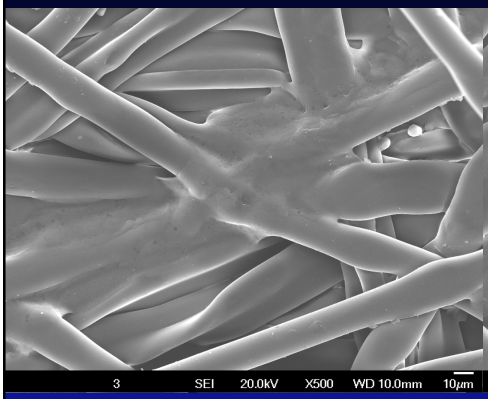
SEM of Membranes (S7:1 layer 280Da , 25psi)

Before Filtration (100x) | After Filtration (100x)

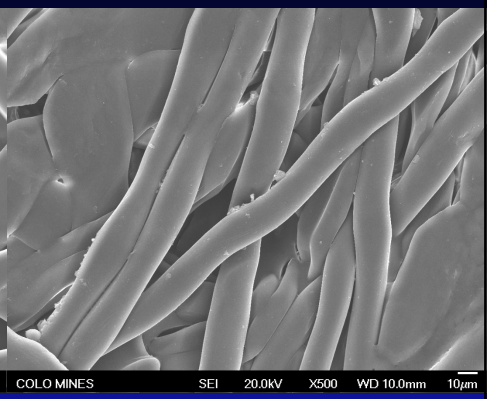


SEM of Membranes (S7:1 layer 280Da , 25psi)

Before Filtration (500x)

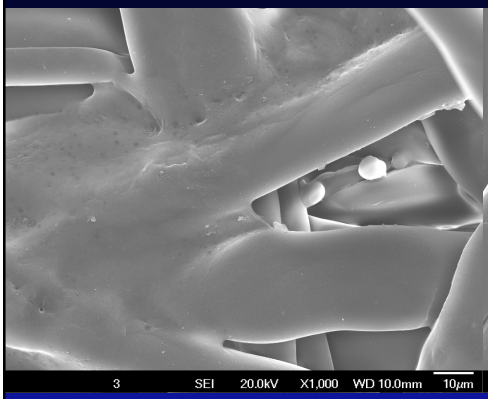


After Filtration (500x)

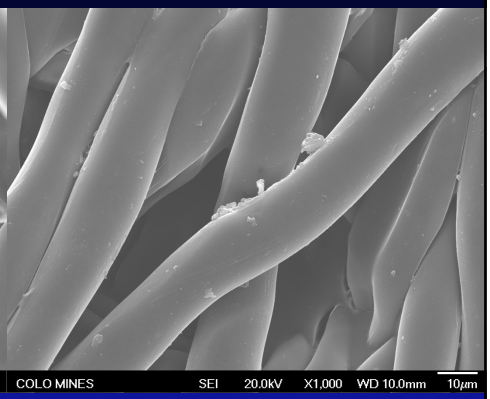


SEM of Membranes (S7:1 layer 280Da , 25psi)

Before Filtration (1000x)

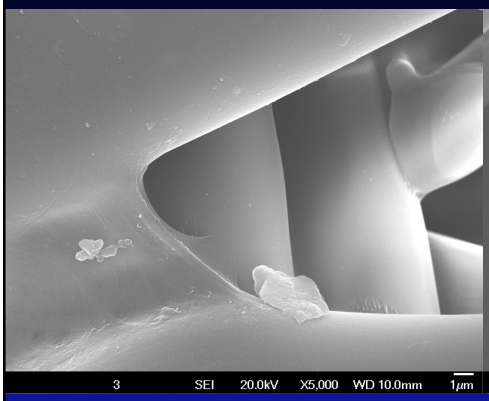


After Filtration (1000x)

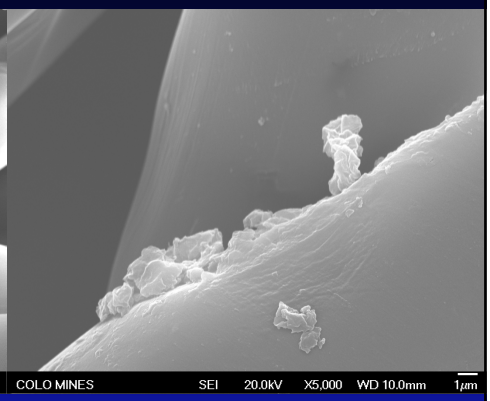


SEM of Membranes (S7:1 layer 280Da, 25psi)

Before Filtration (5000x)

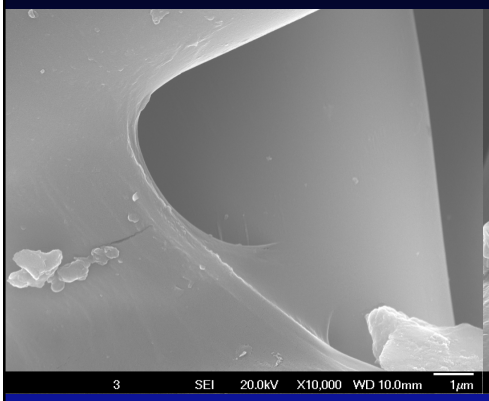


After Filtration (5000x)

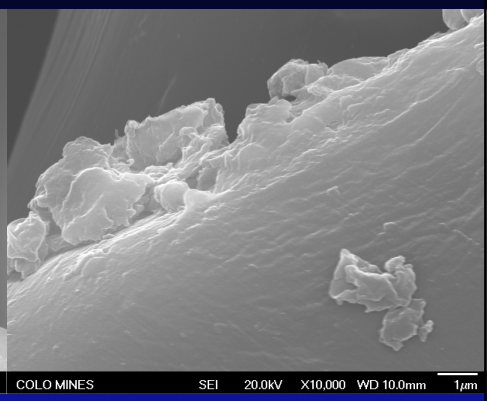


SEM of Membranes (S7:1 layer 280Da, 25psi)

Before Filtration (10kx)



After Filtration (10kx)



CONCLUSIONS

Conclusions

- *No evident filtration effect observed in tests with C6/C12 mixture flowing through 280 Da membranes*
  - *Mixture composition changes fall within experimental errors*
- *Evaporation of lighter component of mixture may affect accurate measurement of mixture composition*
- *Flow process does not change pore structure*
  - *High pressure may compress the membrane but does not change pore structure*

FUTURE WORK

Future Work

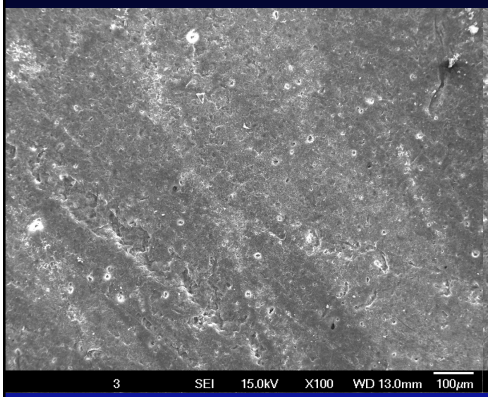
- *Design a set-up that allows multiple/repeat filtration process through membranes within the sealed system to resemble reservoir/core condition*
- *Replace C12 with higher molecular weight hydrocarbon, i.e. C15-18*
- *Search for new membrane material with smaller pore size*
  - *Carbon molecular sieve (<1 nm)*

Thank You!

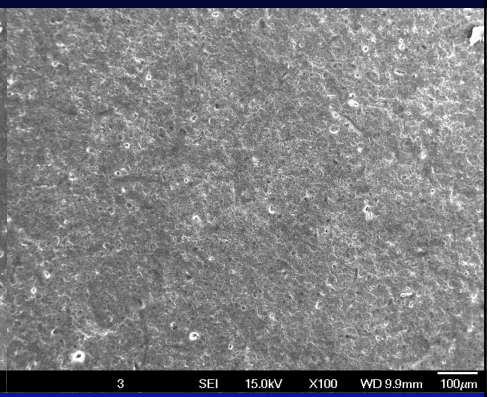
Questions and Suggestions?

SEM of Membranes (1 layer 20nm, 60 psi, 100x)

Before Filtration



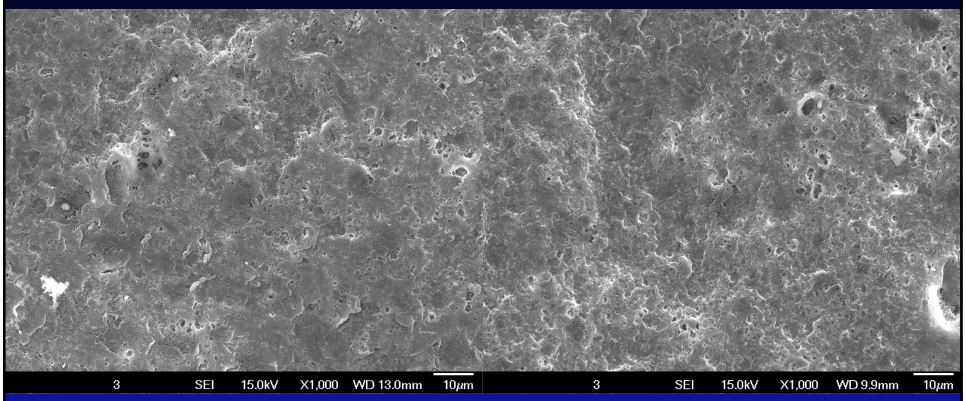
After Filtration



SEM of Membranes (1 layer 20nm, 60 psi, 1000x)

Before Filtration

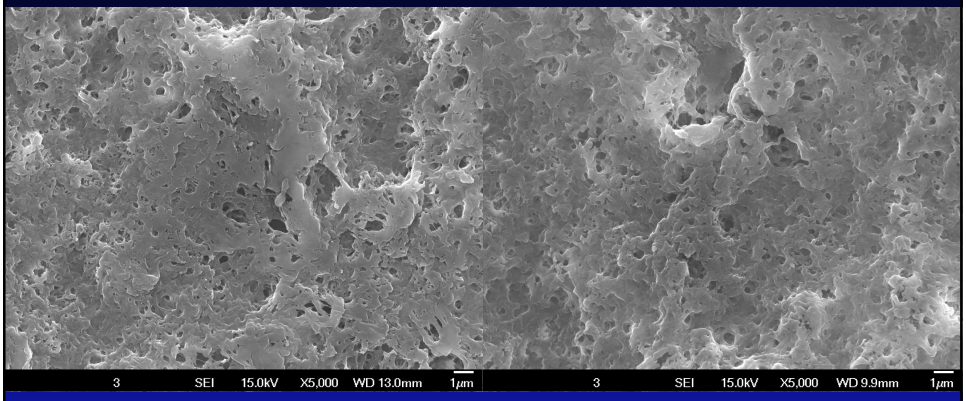
After Filtration



SEM of Membranes (1 layer 20nm, 60 psi, 5000x)

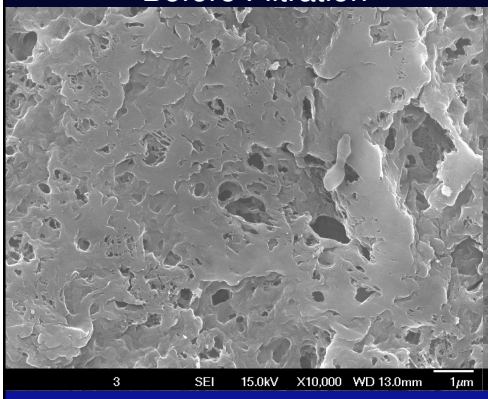
Before Filtration

After Filtration

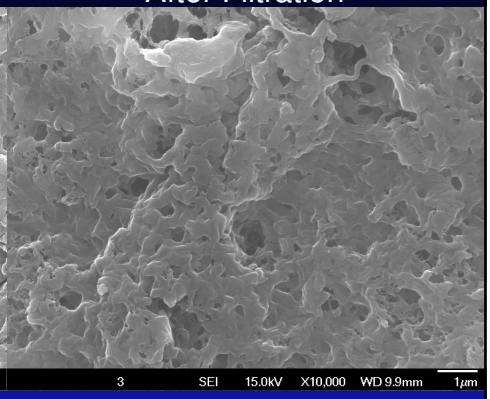


SEM of Membranes (1 layer 20nm, 60 psi, 10kx)

Before Filtration

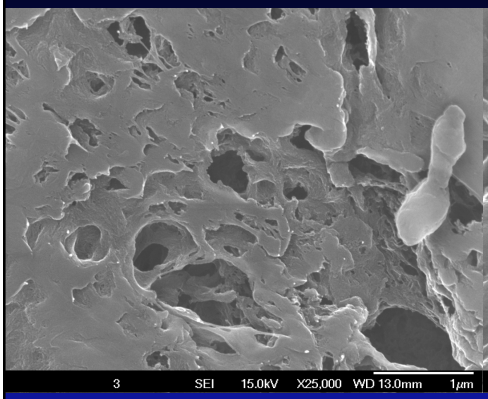


After Filtration

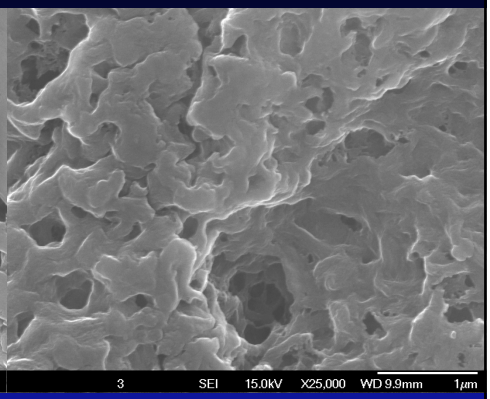


SEM of Membranes (1 layer 20nm, 60 psi, 25kx)

Before Filtration

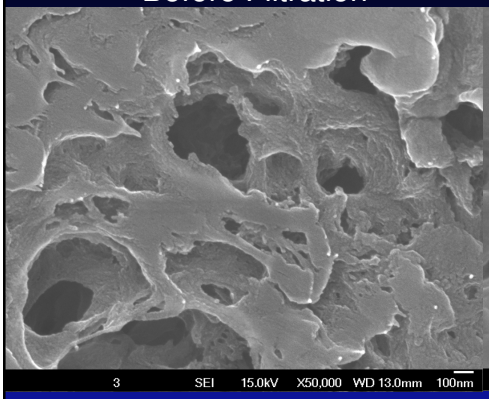


After Filtration



SEM of Membranes (1 layer 20nm, 60 psi, 50kx)

Before Filtration



After Filtration