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Winery Trials on Gas Control Using 3M Liqui-Cel

3M Separation & Purification Sciences
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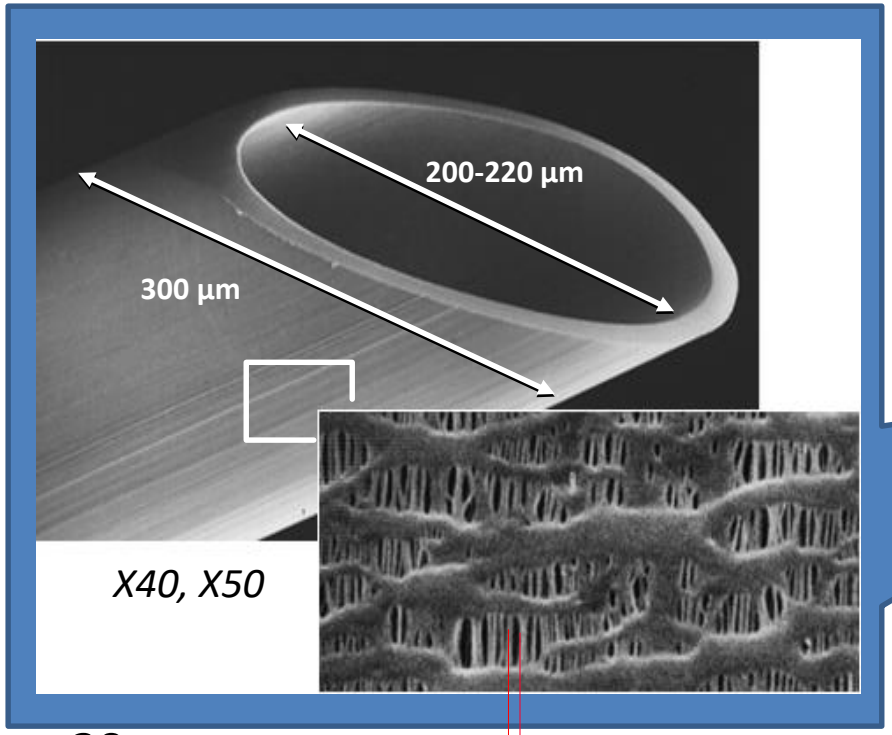
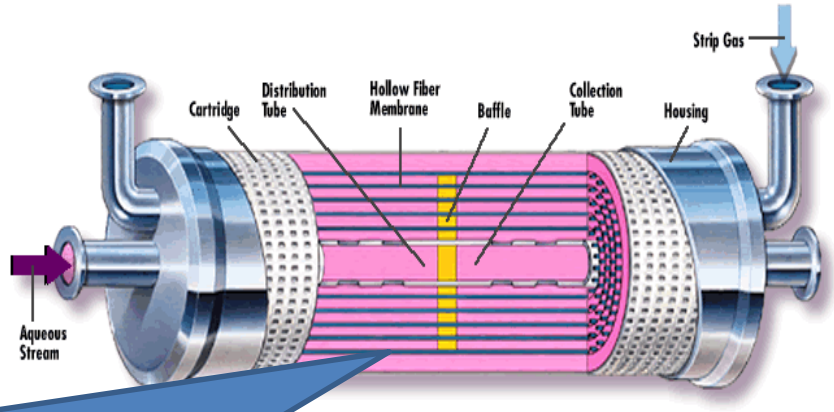
3M Liqui-Cel hollow fiber technology





30 nanometer

<u>Effective</u> Pore Size

3M | Liqui-Cel™ Membrane Contactors

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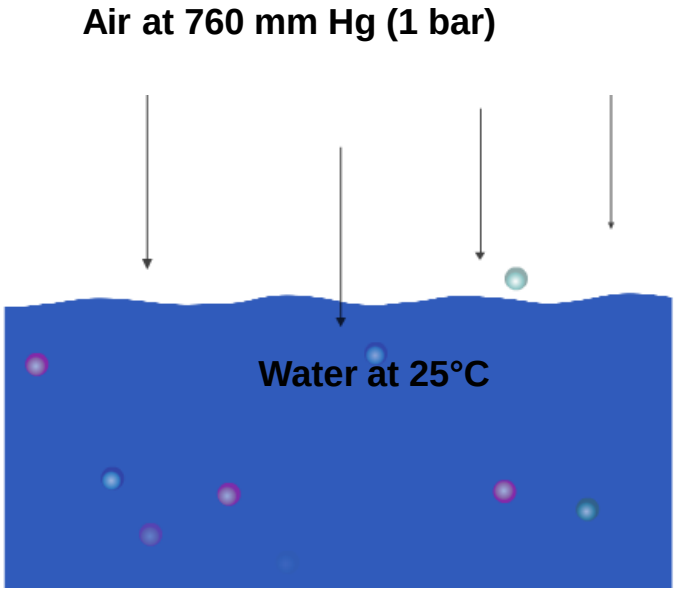
How 3M Liqui-Cel works

- Gas in contact with water will tend to dissolve into water
- The total amount of gas that will dissolve into water is proportional to the gas pressure
- Henry's law (P=Hx)
  - P = partial pressure of the gas in contact with the water
  - H = Henry's proportionality constant of the gas
  - x = concentration of dissolved gas in water (gas solubility in water)



Dissolved oxygen in water = 8.2 ppm
Dissolved nitrogen in water = 13.9 ppm

Henry's Law shows that dissolved gas transfer is related to partial pressures

Gas Solubility in Water (ppm): O_2 (8) < N_2 (14) < Ar (54) < CO_2 (1500)

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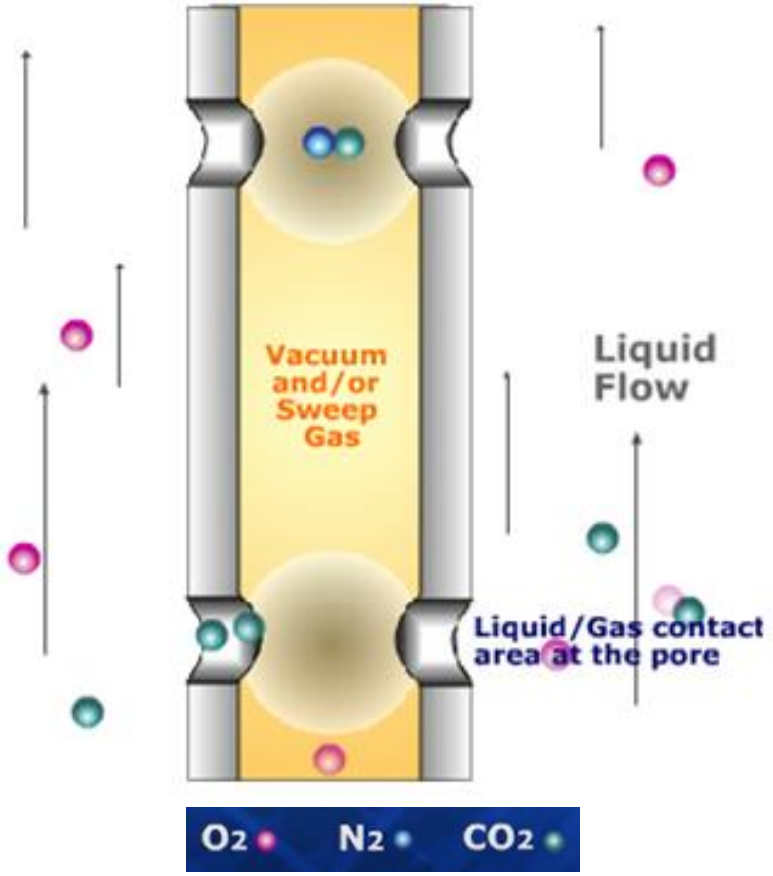
Gas Transfer with 3M Liqui-Cel

- Equilibrium between the liquid and gas phase is offset
- Forms difference between the partial pressure exerted by the gas species in the liquid and the partial pressure of the same gas species in the strip gas or vacuum stream
- This creates a driving force to move gases into or out of the liquid phase with the direction of diffusion from the stream of high partial pressure to the stream of lower partial pressure
- Gas transfer occurs rapidly at the contact point (pore) on the hydrophobic membrane



By changing the partial pressure of the gas we can remove or add dissolved gases to a liquid

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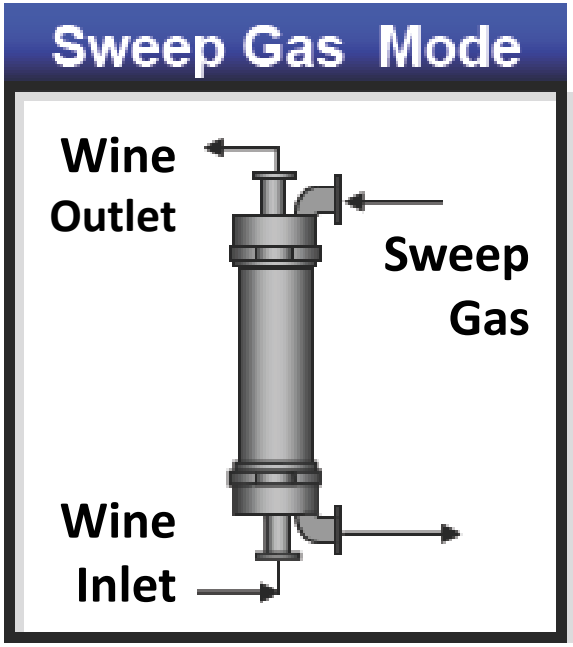
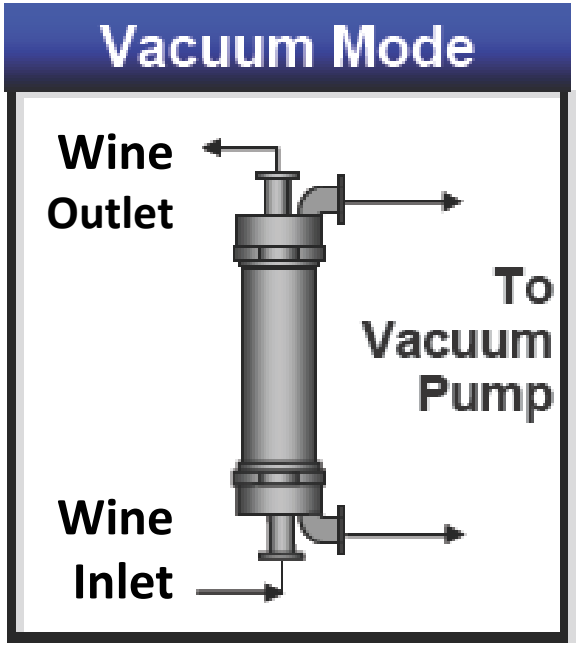
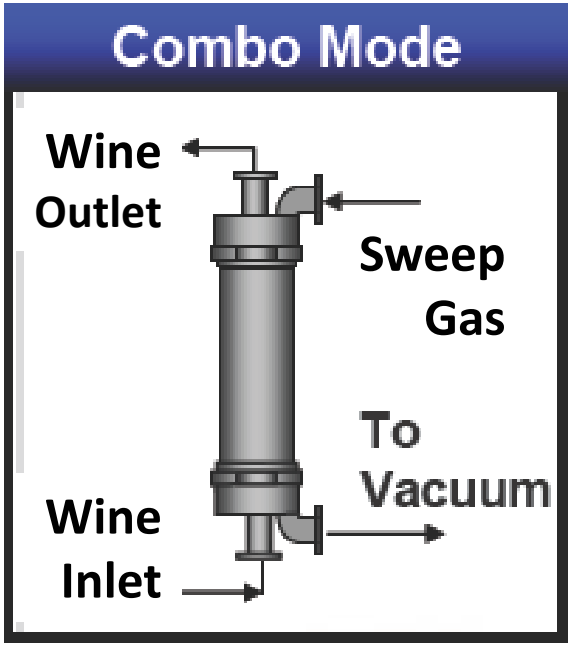
3M Liqui-Cel operation

- **By changing the partial pressure of the gas we can either remove from or dissolve gas into wine**
- **Lower the partial pressure, the gas will be removed from the wine**
- **Increase the partial pressure, the gas will dissolve into the wine**







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Proposed Designs

Flow Rate	10,000 – 30,000 L/hr	2,000 L/hr
Control	CO_2 (0.3 – 2.5 g/L)	O_2 (<10 ppb)
Dim.	800 x 1200 x 1600	600 x 600 x 1600
Gas measurement	Optional	Optional

Winery Test Skid Typical Operating Range

- Carbonation from 1.2 to 1.6g/L at 20m^3/Hr
- Decarbonation from 1.6 to 1.2g/L at 15m^3/Hr
- Deoxygenation while maintaining CO_2 level
  - NOTE: with a single Liqui-Cel contactor, it is only possible to control one gas at a time so maintaining CO_2 while stripping O_2 can be done (60-80%), but cannot set an O_2 value

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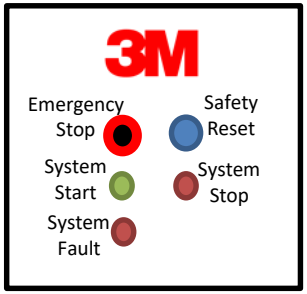
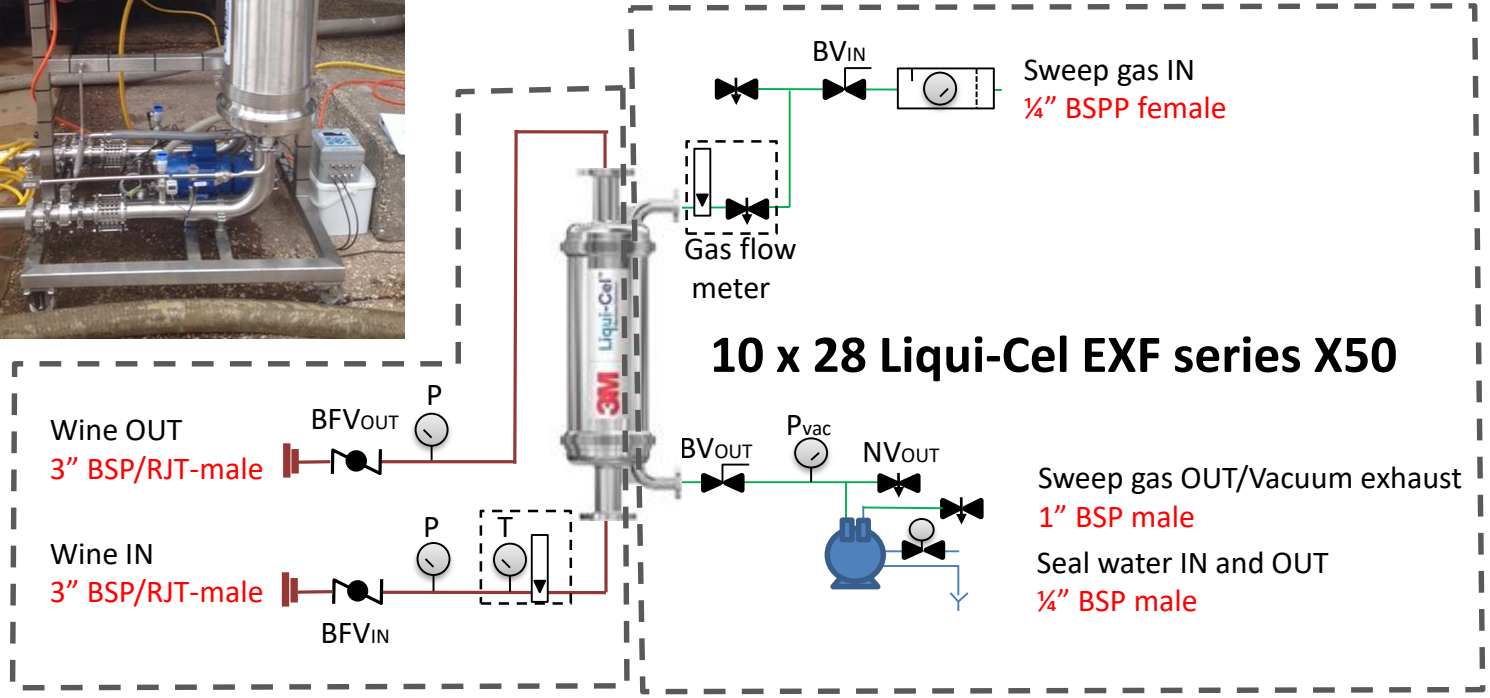
3M ANZ Liqui-Cel Test Skid



415V (3 phase), 50Hz
Max 5A

5 pin plug

CONTROL PANEL



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Trial Stages

1. Installation

- Power supply
- Vacuum seal water
- Wine hose
- Wine pump
- Rinse tank (200L)
- Gas supply (CO2)

2. Demo with water

- Filtered water (at least 5µm)
- Initial rinsing of skid
- Demo skid operation at recirculated loop to a rinse tank

3. CIP

- Filtered water rinse
- Alkaline recirculation
- Acid recirculation
- Filtered water rinse

4. Wine gas control

- Recirculated loop for parameter adjustment
- Single pass with monitoring and data gathering
- Training and handing over operation to customer

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Degassing & carbonation wine trials

Dataset 1

Wine Style	Operation	Wine flow rate [m3/Hr]	Mode	Sweep Gas	Target CO2 [g/L]	Actual INLET CO2 [g/L]	Actual OUTLET CO2 [g/L]	Temperature [°C]	Processed Volume [kL]	Process Time [Hr]
Sav Blanc	Decarbonation	20	Combo	N2	0.7	1.2	0.77	8.4		
Sav Blanc	Decarbonation	15	Combo	CO2	0.7	1.2	0.77	8.8		
Sav Blanc	Carbonation	15	Sweep	CO2	1.2	0.62	1.2	9.4	19.1	1.27
Sav Blanc	Decarbonation	13	Combo	CO2	0.6	1.2	0.61	8.4	19.1	1.47
Chardonnay	Carbonation	15	Sweep	CO2	1.2	0.68	1.21	8.1	19.8	1.32
Chardonnay	Decarbonation	13	Combo	CO2	0.6	1.21	0.64	8.1	19.8	1.52
Moscato	Carbonation	29	Sweep	CO2		1.2	7.2	9.6		

Dataset 2

Wine Style	Operation	Wine flow rate [m3/Hr]	Mode	Sweep Gas	Target O2 [ppm]	Target CO2 [g/L]	Actual INLET O2 [ppm]	Actual INLET CO2 [g/L]	Actual OUTLET O2 [ppm]	Actual OUTLET CO2 [g/L]	Temp [°C]
Pink Moscato	Deoxy-Carbo	8	Sweep	CO2	1	3	2.5	0.8	0.439	3.12	3.4
Pink Moscato	Deoxygenation	8	Vacuum	None			2.5	0.8	0.272	0.098	3.4
Prosecco	Carbonation	5	Sweep	CO2		11-11.9		6.27		11.3	-1.5
Sparkling	Carbonation	5	Sweep	CO2		11-11.9		4.7		**17.2**	-3.2
Sparkling	Carbonation	4	Sweep	CO2				?		5.64	5
Sparkling	Carbonation	6.5	Sweep	CO2				5.64		8.89	2.5

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Sparging vs Liqui-Cel wine trial

Wine style	White wine		
Initial dCO2 [g/L]	0.31		
Target dCO2 [g/L]	0.72		
Dissolved Gas Management	Sparging	Liqui-Cel	Sparging Estimates
Wine Flow rate [L/hr]	21000	21000	
Volume [L]	45000	8400	45000
Duration [min]	128.6	24	
CO2 used [kg]	24	3.84	65.6
Final dCO2 [g/L]	0.46	0.72	0.72
Cost of CO2 used	9.51	1.52	25.99
CO2 cost/L wine	0.00021	0.00018	0.00058

Approximately12% over the theoretical requirement

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Sensory trial – Duo Trio

Degassing with sparger vs 3M Liqui-Cel gas contactor

Assessor:	Set:	Reference sample	Different sample #	Selected different #	Correct selection
A	1	Liqui-Cel	2	1	n
	2	N2 sparger	2	1	n
	3	N2 sparger	2	2	y
	4	Liqui-Cel	2	2	y
	5	Liqui-Cel	2	1	n
	6	N2 sparger	2	2	y
B	1	Liqui-Cel	2	1	n
	2	N2 sparger	2	2	y
	3	N2 sparger	2	2	y
	4	Liqui-Cel	2	2	y
	5	Liqui-Cel	2	2	y
	6	N2	2	2	y
C	1	Liqui-Cel	2	2	y
	2	N2 sparger	2	2	y
	3	N2 sparger	2	2	y
	4	Liqui-Cel	2	1	n
	5	Liqui-Cel	2	1	n
	6	N2 sparger	2	2	y
D	1	Liqui-Cel	2	2	y
	2	N2 sparger	2	1	n
	3	N2 sparger	2	2	y
	4	Liqui-Cel	2	1	n
	5	Liqui-Cel	2	1	n
	6	N2 sparger	2	2	y

Assessor:	Set:	Reference sample	Different sample #	Selected different #	Correct selection
E	1	Liqui-Cel	2	2	y
	2	N2 sparger	2	2	y
	3	N2 sparger	2	2	y
	4	Liqui-Cel	2	1	n
	5	Liqui-Cel	2	1	n
	6	N2 sparger	2	1	n
F	1	Liqui-Cel	2	1	n
	2	N2 sparger	2	2	y
	3	N2 sparger	2	1	n
	4	Liqui-Cel	2	1	n
	5	Liqui-Cel	2	2	y
	6	N2 sparger	2	1	n
G	1	Liqui-Cel	2	2	y
	2	N2 sparger	2	2	y
	3	N2 sparger	2	1	n
	4	Liqui-Cel	2	1	n
	5	Liqui-Cel	2	1	n
	6	N2 sparger	2	1	n

Wine Style: **Sauvignon Blanc**

Total Assessed: **42** Total Correct: **22**

27 Needed for Significant at 95% Level

Difference between treatment: **Not significant**

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Sensory trial – Triangle test

Carbonation with sparger vs 3M Liqui-Cel gas contactor

Wine Style: **Prosecco carbonated up to 6vol CO2 (11.8g/L)**

First Triangle Test (March 2018)
Total Assessed: **18 (6 tasters x 3 brackets per taster)**
Total Correct: **8 correctly picked the different sample**
10 Correct responses Required for Significant Difference at 95% Level

Second Triangle Test (June 2018)
Total Assessed: **24 (8 tasters x 3 brackets per taster)**
Total Correct: **5 correctly picked the different sample**
13 Correct responses Required for Significant Difference at 95% Level

Difference between treatments: **Not significant (both First and Second Triangle Tests)**

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Key findings

Precise control of dissolved gas concentration

- Steady and consistent dissolved gas concentration at output
- Adjustment at start-up, no trial and error

Efficient transfer/removal of gas

- Reduction of process time (less rework, no temp adjustment) = ease of operation
- Minimise gas breakout (no large sized bubbles, no foaming on tanks)

No need for wine temperature adjustment (typical when degassing with sparger) **= less wine handling, less energy use**

No negative impact on sensory characteristic of wine = MOST IMPORTANT CRITERIA

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Interested?

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