

Non-dispersive diffusion for nitrogenation

The non-dispersive diffusion system features a Liqui-Cel®, Extra-Flow™ Membrane Contactor manufactured by Celgard, Inc. of Charlotte, NC. The high-efficiency contactor features a hollow-fiber contact medium and has been used to transfer a variety of dissolved gases, to or from an aqueous stream, in a variety of applications in the food and beverage, semiconductor, power, chemical, and pharmaceutical industries.

At the Mansfield Brewery, the market-driven purpose of nitrogen introduction was to lower CO₂ content, which was necessary to create a "smooth" beer – a "keg" beer that would mimic "cask-conditioned" beers, but without losing the "head" characteristic of keg beers.

Mike Cleator, the brewery's chief engineer, explained that dispersive direct-injection technology already available for nitrogen introduction was considered disadvantageous. Direct injection would have introduced highly undesirable turbulence that would have knocked CO₂ out of solution, because in order to make the nitrogen dissolve with any speed, pressure would have to be raised up to 8 – 9 bar (132 psi). With the Celgard technology, nitrogen could be introduced at low pressure – typically 30 – 45 psi – a level that is actually lower than the 60 psi pressure needed to move the beer around.

According to Mike Cleator, capital cost savings were a significant advantage for the installed system, which has been performing well.

The cost for the non-dispersive nitrogen diffusion system, consisting of the Celgard membrane, an Orbisphere 2-channel CO₂/N₂ meter, a control panel, and a membrane cleaning tank with pump & heater, came in at about \$64,000. By comparison, off-the-shelf cost for a dispersive direct-injection system would have been about \$144,000. The system has now been operating successfully since May, 1997 overall, and since December, 1998 with a revised membrane cleaning operation that has boosted production levels.

Operations management at Mansfield Brewery Company here reports it has successfully applied a non-dispersive diffusion system to introduce nitrogen into its beer-making processes. The move avoided the need for a dispersive direct injection alternative that would have mandated much higher cost, caused highly undesirable turbulence, and offered significantly less control of dissolved gas concentrations.

In addition, there have been important process control advantages.

The nitrogenation pressure is very easy to control, there are no extra pumps necessary to raise the pressure, and no valves to then slowly reduce it. Moreover, avoiding the reduction of beer pressure over valves takes away the associated risk of inducing CO₂ breakout. Overall, as the brewery's chief engineer stated, this method offers considerably more flexibility as a processing tool than straightforward injection would, especially regarding control of CO₂ content.

Cleator elaborated that he cannot allow water and CO₂ to build up on the nitrogen side of the membrane. Because of CO₂ and nitrogen partial pressures, he needs to keep increasing the nitrogen, but cannot exceed the beer pressure. So he has a continuous purge of gas from the lumen side of the membrane that removes the water vapor and sweeps out CO₂.

Per Henry's Law, dissolved oxygen content of the beer is reduced as it goes through the membrane. With mixed gas placed on the membrane, Henry's Law also allows adjustment of CO₂ content at the same time as nitrogenation. The amount of CO₂ that gets removed is proportional to the amount of nitrogen sweep gas purged out.

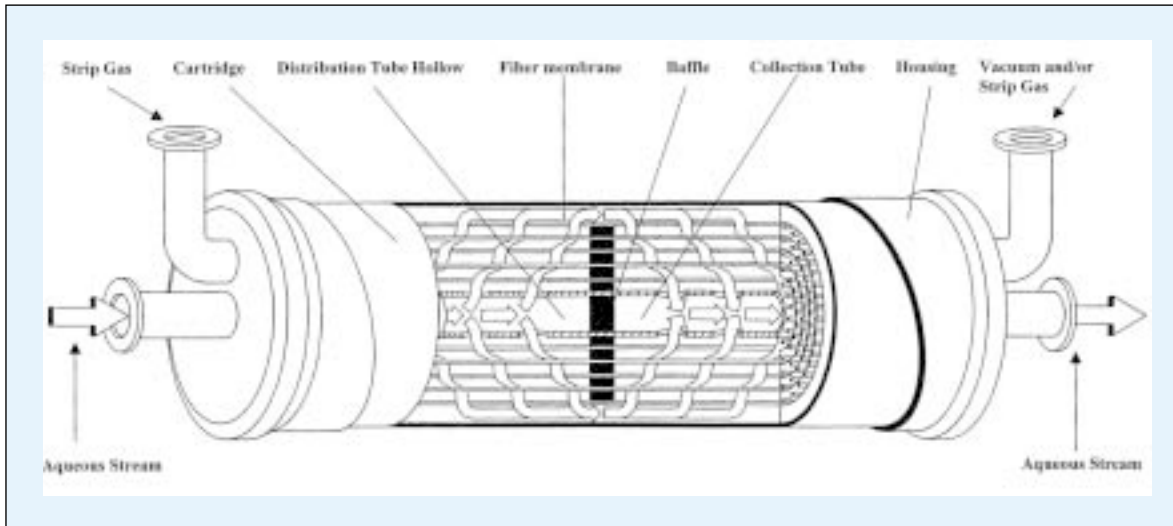
If the beer is too high in CO₂, the traditional remedy is to purge it by injecting nitrogen, thereby shocking it out of solution. But this is, as Mike Cleator put it, "very inefficient, wasting nitrogen, and also very messy, with lots of froth coming out of the vent and all over the floor, typically at the same time the Chairman is walking around." Alternatively, the nitrogen sweep rate is just increased, increasing CO₂ through the membrane, and reducing CO₂ concentration to within specification.

Conversely, a lower CO₂ level can be raised to within specification by introducing a mix of CO₂ and nitrogen on the gas side of the membrane. This allows for nitrogenation and carbonation at the same time, with typical CO₂ concentration at 2 – 2.5 g/l.

Regarding membrane cleaning, which is needed after each use, the cycle includes water rinse to remove beer residue, followed by 2% NaOH for 1/2 hr., then cold water rinse for 10 min., and finally 600 ppm sulfite KMS (potassium meta-bisulfite) sterilizing solution, which is circulated and then left full on the membrane until the next use.

The membrane had to be left full of sterilizing solution after a run, and rinsed out before the next run. But this could be automated, with push-button initiation of the cleaning sequence beginning with water entering to flush out the sterilizing solution.

Initially, the brewer wanted to use the 2% NaOH at 10 – 12 °C (40 – 54 °F), instead of at 30 °C (86 °F) as recommended by Celgard, because of concern about the presence of hot caustic. But that resulted in some liquid wetting through into the gas, or lumen, side of the membrane. Consequent reduction in membrane performance was realized as lower nitrogen in the beer. In order to achieve the 30 – 45 ppm nitrogen level desired, it was necessary to reduce the beer flow rate from 120 barrels/hr to 50 barrels/hr.



Membrane contactor cutaway: Liqui-Cel® Extra-Flow™ membrane contactor

Cleator reports that when he started using the NaOH at the higher temperature, he readily raised production up to 120 - 130 bbl/hr, with frequency of cleaning not a problem.

Cleaning is done 4x/week only because in the brewery different types of beer are running. Upon cleaning, the membrane has always returned to its original installed condition and performance, as Mike Cleator stated. It does now take 2 hrs. to heat the NaOH vs. 1 hr previously. But should this later be deemed a problem, the current tank immersion heater could simply be replaced with a steam heater.

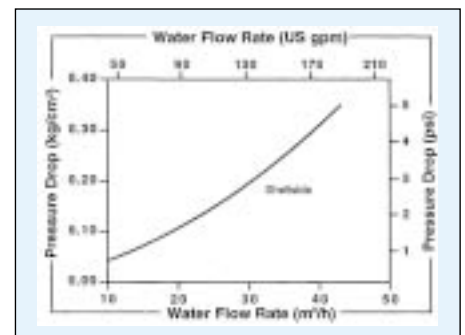
Regarding operating costs, Cleator said "it's all in the detergent," which he notes is relatively inexpensive because it must be a chlorine-free crude form. Before establishing proper cleaning technique, prefiltration costs had been high due to changing filters once a week, but they now are changed only every 6 - 8 weeks. Each change of the 10-

micron pore size filters costs \$480, and lasts for 8000 barrels of production.

As for ease of operation, special expertise is not required. When a runoff is started, the operators stand by to make sure it reaches a steady state within 5 - 10 min., then they can go away and do other things and check about every half an hour. Further automation is not needed. For very large production, to include this cleaning operation in the automation, only a control valve linked to the output of the now manually-set nitrogen meter would be necessary.

Celgard's Liqui-Cel® Extra-Flow™ membrane contactors have been performing commercially since 1991. The patented design contains microporous polypropylene hollow fibers knitted into an array that is wound around a distribution tube with a central baffle. The hollow fibers are arranged in a uniform open packing, allowing for greater flow capacity and utilization of the total membrane surface area.

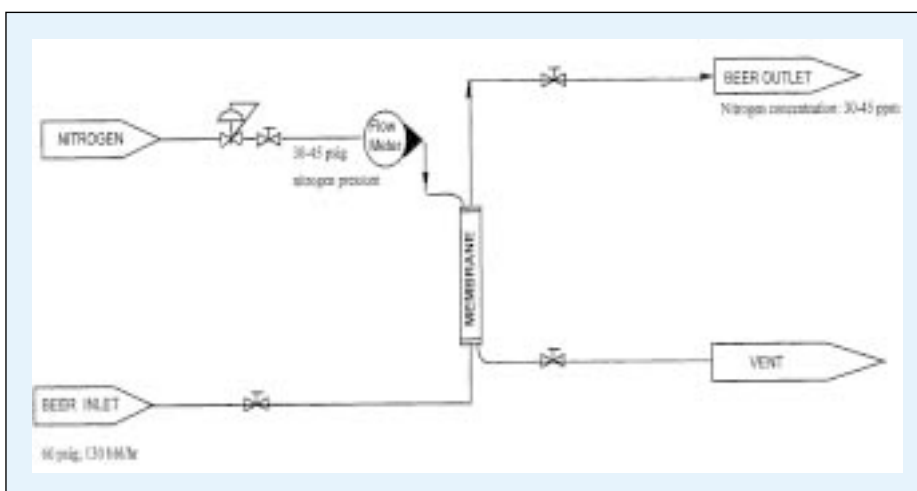
During operation, an aqueous liquid flows over the outside of the hollow fibers, with the patented design forcing it radially across the array. A strip gas or vacuum, separately or in combination, is applied on the inside of the hollow fibers. Because of its hydrophobic nature, the membrane acts as an inert support, allowing intimate contact between gas and liquid phases without dispersion.



Typical low pressure drop curve for Celgard LLC's Liqui-Cel® Extra-Flow™ membrane contactor, representing nominal values generated using water at 20°C (68°F). Characteristics may change under different operating conditions

The interface is immobilized at the pore by applying a higher pressure to the aqueous stream relative to the gas stream. As the aqueous stream flows over the outside of the fibers, the strip gas or vacuum flows countercurrent inside the hollow fibers. The result is fast diffusive transfer of dissolved gases from or to the liquid phase.

In addition to nitrogenation, the membrane has been used for ultrapure water deoxygenation and decarbonation; boiler feed water degassing; and process water deaeration and carbonation. ■



Schematic process diagram: Non-dispersive diffusion system featuring high-efficiency membrane contactor was utilized instead of dispersive direct injection to introduce nitrogen into the beer-making process at Mansfield Brewery Company