Compact Membrane Systems (CMS) applications in solvent dehydration

Overview

Solvents are required for many chemical, industrial, and pharmaceutical processes for a variety of process steps including synthetic chemistry, removal of intermediates, purifying final products for packaging, clean-up of machinery, and more. While some solvents are recovered and reused, others are not due to the cost or challenge in recovering them to the necessary purity. The solvent recovery process can still be expensive or cumbersome, involving multiple distillation steps and large columns for components with similar volatilities, and large quantities of heat to drive evaporation. In addition, as pharmaceutical manufacturing for small molecules is moving more towards flow chemistry or continuous manufacturing (as opposed to batch), it can be logistically difficult

Compact Membrane Systems (CMS) offers a new, membrane-based separation technology that is highly effective at dehydration and separation, drying solvents to 99.5+% purity. CMS technology is particularly effective in breaking azeotropic solvent-water mixtures, situations where water is the minor component and it is dissolved and/or dispersed, and in close boiling point mixtures. CMS technology is cost-effective and easy to use, enabling economic recovery of most solvents to very dry levels.

CMS uses perfluoromembranes with exceptional chemical and thermal stability and gas transport properties, which provide performance and cost superiority to conventional alternatives that require multiple steps and heat input for evaporation.

The Current Challenges

Currently, few solutions exist for drying solvents to very dry levels. For example, isopropyl alcohol, ethanol and tetrahydrofuran all form azeotropes, making further dehydration nearly impossible for conventional, distillation based technologies. In these situations, consumers regularly and repeatedly purchase virgin solvent, sending spent solvent to incineration as toxic waste. This solution is both costly and environmentally unappealing. Alternatively, manufacturers may reuse solvent that has taken on water, but pay a penalty in terms of performance.

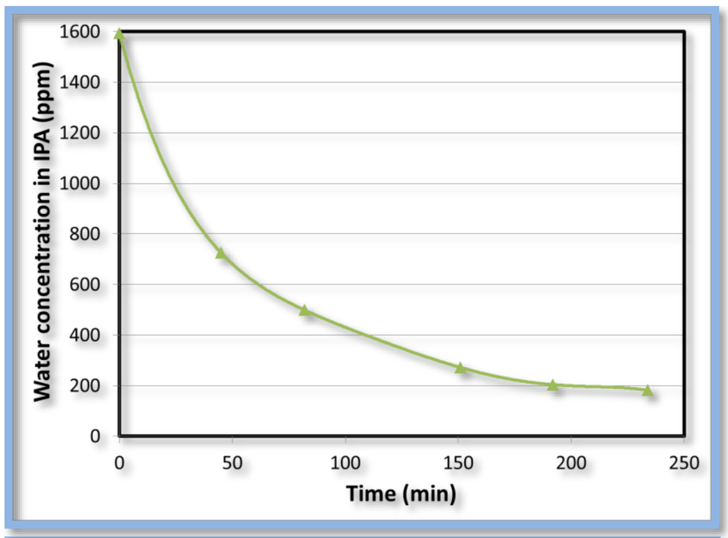
Pharmaceutical manufacturers that are interested in moving towards flow chemistry are often deterred by the amount of solvent needed. Recycling solvent on a large scale is more easily done with membranes than traditional distillation technology. Membranes have the added advantage of lower operating temperatures to avoid the degradation of sensitive APIs . Utilizing a membrane solution also negates the need for entrainers, molecular sieves, or pressure swing distillation.

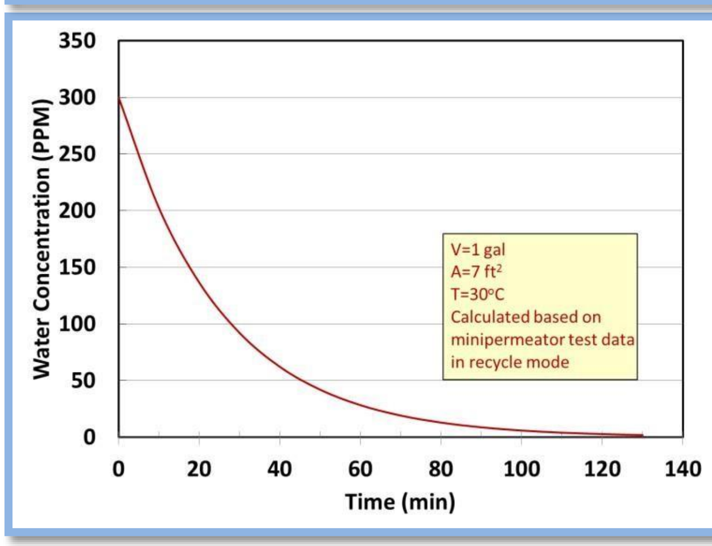
In the face of rising solvent costs, increased interest in flow chemistry, the benefits of green chemistry, and greater attention to risks in managing and transporting hazardous waste, recovering spent solvents is more critical than in the past.

Technology Platform, Performance, and Value

Utilizing novel technologies that address the shortcomings of distillation based approaches can be an important component of reducing production and recovery costs, increasing solvent purity, reducing environmental impact, and ensuring high quality end product.

Compact Membrane Systems' (CMS) membrane-based technology is efficient and cost-effective at dehydrating and recovering a number of solvents to very high levels of purity. CMS membranes can perform a valuable service: offering a cost-effective way to conduct difficult separations and/or as a finishing step after distillation to enhance purity to high levels. Using mass transfer principles, CMS membranes can reduce water to below 10 ppm in immiscible solvent-water systems (e.g. xylene, hexane, toluene) without the use of entrainers to break multiple distillation steps. In miscible solvent-water systems (e.g. IPA, EtOH, THF) the membranes can decrease water content to below 500 ppm and often times even lower. Systems are easy to use and can be adapted to different applications, to meet customer need for specific purity and recovery levels. Example results for IPA and toluene drying processes, at right, show substantial reductions in water content. CMS has also worked with Pfizer on membranes for the continuous production of anhydrous tert-butyl hydroperoxide in nonane. The resulting paper has been published and can be found on the CMS website.





CMS technology has proven effective at drying the following solvents:

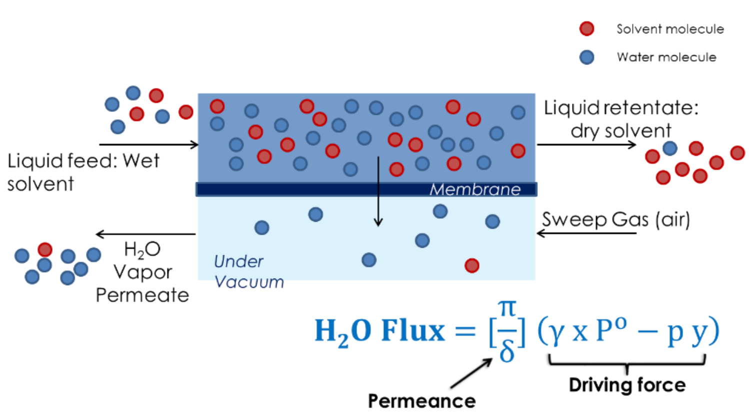
Common Pharmaceutical and Industrial Solvents		
Solvent	**Normal Boiling Points (°C)**	**CMS technology effective?**
p-Xylene	139	y
Ethylbenzene	136	y
Chlorobenzene	131	y
Toluene	111	y
N-Hexane	68	y
Cyclohexane	81	y
N-Butyl Alcohol	118	y
Isopropyl Alcohol	82	y
THF	66	y
Methyl Tert-Butyl Ether	55	y
Methyl Isobutyl Ketone	117	y
EtOH	78	y
Acetonitrile	82	Low selectivity
Methanol	65	Low selectivity
Dichloromethane	40	Not compatible
Chloroform	61	Not compatible

Boiling Points and Solubilities were obtained from http://macro.lsu.edu/HowTo/solvents.htm
*20 C , **25 C

How it works

CMS technology works by pervaporation. Pervaporation, in its simplest form, is an energy efficient combination of membrane permeation and evaporation. Pervaporation involves the separation of two or more components across a membrane by differing rates of diffusion through a thin polymer film and an evaporative phase change comparable to a simple flash step[ii].

The diagram below is a simplified view of the separation. A liquid feed is fed to a membrane and vacuum is pulled on the backside of the membrane. The material that doesn't travel through the membrane is called the retentate while the material that flows through is called the permeate.



The water flux equation as shown in the diagram above contains several variables that determine the driving force as well as permeance:

x = water feed mole fraction
y = water permeate mole fraction
π = water permeability
γ = water activity coefficient
P^o = water vapor pressure
δ = membrane thickness
p = vacuum pressure

The driving force (and therefore water flux) can be increased through increasing the temperature or decreasing the permeate side pressure.

Pervaporation works efficiently in recycle mode or continuous one-pass mode. A vapor pressure gradient is used to allow one component to preferentially permeate across the membrane. A vacuum applied to the permeate side is coupled with the immediate condensation of the permeated vapors. Pervaporation is typically suited to separating a minor component of a liquid mixture, thus high selectivity through the membrane isessential.

The liquid feed to the membrane is heated once to operating temperature and doesn't have tobe cooled until the dehydration is completed. The only heating required during the process is that needed for the evaporation of the permeate and for making up the heat losses from the system to maintain the feed at operation temperature.

CMS membrane systems are chemically and thermally resistant and suitable for a range of dehydration applications, including oil, solvents, and other chemicals. CMS membranes are most effective where water is the minor component and processed volumes are less than 5000 gallons per day. They can remove even small quantities of water, to provide product that is 99.9+% free of water and other components. CMS membrane modules can be used in harsh environments as they are chemically and thermally resistant, and work very well with viscous fluids (oils and lubricants). They also resist fouling and thus are easier to operate and maintain than alternative dewatering systems. T

Operation of a membrane cartridge

Membranes can be operated in either recycle mode or single pass continuous mode. CMS produces two standard size cartridges with smaller systems being available on a custom basis. The membranes can be expected to have a lifetime of 12-24 months and should be protected with a particle filter (<7 µm) prior to the feed entering the cartridge. Unless otherwise requested, cartridges are provided in a stainless steel housing.

2” Cartridge operation details

Property	Value
Flow range	0.1 - 4 GPM
Maximum feed pressure	60 psig
Maximum temperature	80C
Housing dimensions	~13” long, ~2” diameter

4” Cartridge operation details

Property	Value
Flow range	1-8 GPM
Maximum temperature	80C
Maximum feed pressure	50 psig
Housing dimensions	~20.5” long, ~5.5” diameter

About Compact Membrane Systems (CMS)

CMS is an advanced materials company, turning chemistry into sustainability with superior products for advanced chemical separations. CMS perfluoropolymers are compatible with a wide range of chemicals, are resistant to surface fouling, and perform well at high temperature.

The CMS platform is in commercial use in a range of applications and industries, showing long term performance over thousands of weeks of continuous use in power generation, nuclear power plants, marine vessels, wind turbines, mines, paper mills, and more.

CMS has a strong patent portfolio that includes 15 issued patents, and seven pending applications. The portfolio includes composition of matter patents as well as CMS perfluoromembrane patents for removing small molecules from liquids.

[i] EPA-HQ-RCRA-2010-0742

[ii] http://www.cheresources.com/content/articles/separation-technology/pervaporation-an-overiew