

## Case Study : An Energy efficient way to retrieve THF from a waste stream

### Introduction

PERVATECH BV established in 1999 is a leading company in membrane technology. Based on the belief that we have an obligation towards future generations to preserve Earth's resources we develop products and services that enable customers to innovate their products and production processes towards lower energy consumption, less waste and higher quality. We produce membranes, membrane modules and systems for pervaporation and vapour permeation applications.

In this case study we present an energy efficient method to reduce THF rich waste streams and recover as much THF as possible, thereby lowering the replacement cost that comes with the purchase of new clean solvent.

### Case Study

THF is a popular solvent in the production of poly(tetramethylene ether)glycol (PTMEG) which is primarily used for the production of spandex. Other main applications for THF are as an industrial solvent of varnishes and PVC and in the laboratory when a moderately higher-boiling ethereal solvent is required.



Figure 1: THF

THF can be recycled by means of distillation. With this process separating water from THF becomes increasingly difficult with lower water concentrations due to the high reflux and the large number of stages required. A McCabe-Thiele diagram of the complete dehydration of THF from 20 wt.% ( $\approx 50$  mole%) water by means of distillation is depicted in figure 2.

For the dehydration/purification of THF, alternative solutions are possible. One of these solutions is the combination of distillation with pervaporation. Such a hybrid process offers advantages over the stand alone distillation process in terms of energy efficiency. A McCabe-Thiele diagram of the distillation of THF from 20 wt.% water ( $\approx 50$  mole%) to a residual water concentration of 5 wt.% (17.4 mole%) is depicted in figure 3. This diagram shows clearly that the reflux and the number of stages can be reduced considerably when coupled to a pervaporation process which can dehydrate the THF further up to specifications. This hybrid process depicted in figure 4 shows that the top product of the distillation column is sent to the pervaporation process. The permeate -rich in water- is sent back to the distillation column to make sure no organics are lost in the process.

A further alternative solution is the complete dehydration of THF by pervaporation; depending on the size and the water content of the stream this can be an economic option.

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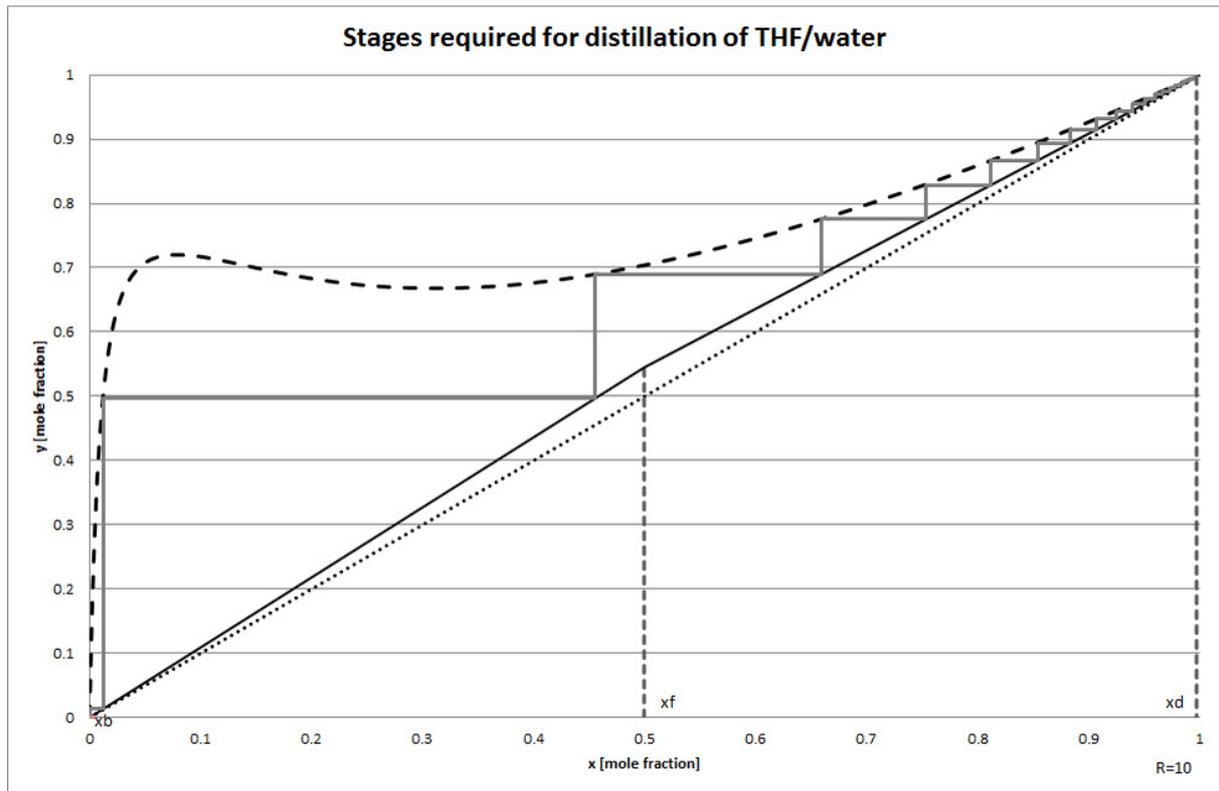
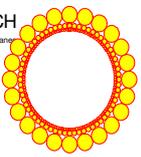


Figure 2: McCabe-Thiele diagram for the complete dehydration of THF

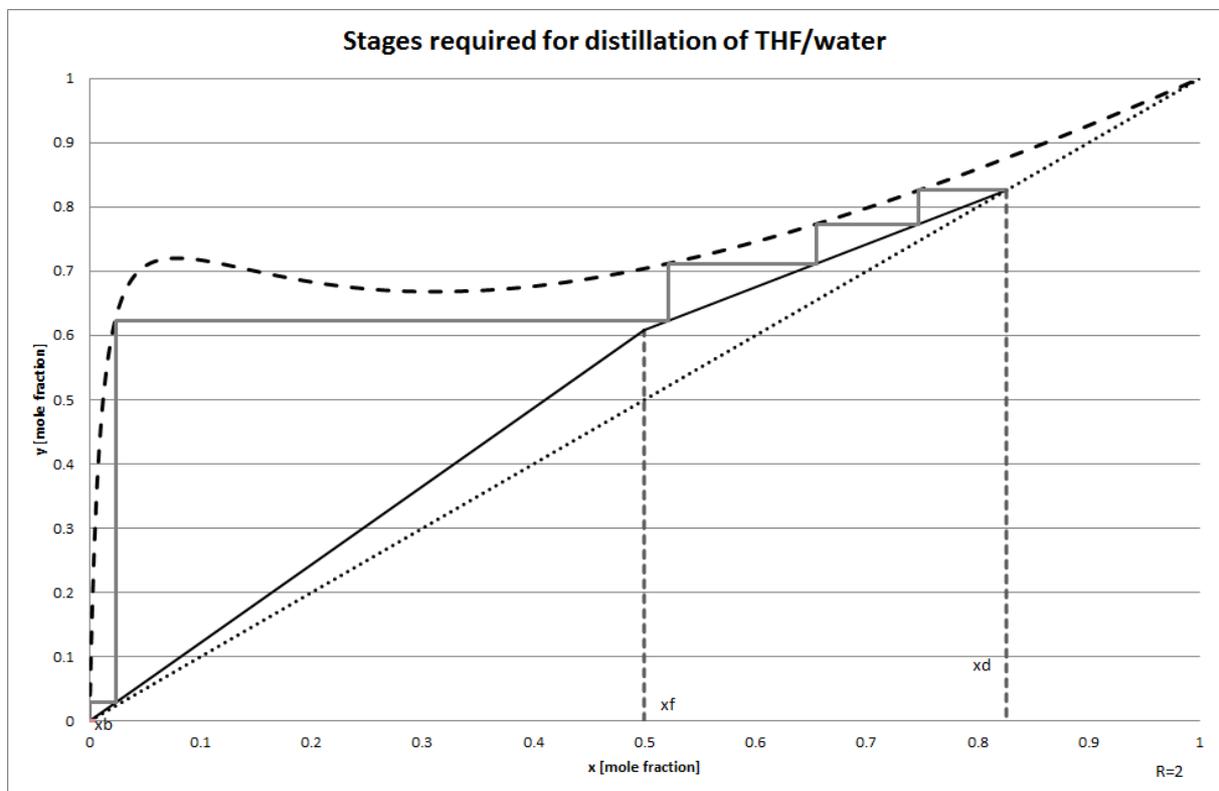
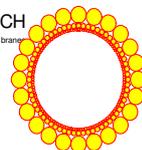


Figure 3: McCabe-Thiele diagram for the dehydration of THF to 5wt% (17.4 mole%) residual water



A combined distillation / pervaporation process for the dehydration of 10,000 kg/day THF was considered. The results obtained show the potential of this environment friendly solution. The mixture is first distilled to a relatively low water concentration of 5 wt.%. Subsequently the THF/water mixture is fed into the pervaporation module and dehydrated to the desired residual water concentration of 0.1 wt.%. The permeate is fed back to the distillation column where the traces of THF that permeate with the water are removed from the water (figure 4). The required amount of membrane surface was calculated based on experiments with Pervatech Hybrid Silica membranes. The results are shown in table 1.

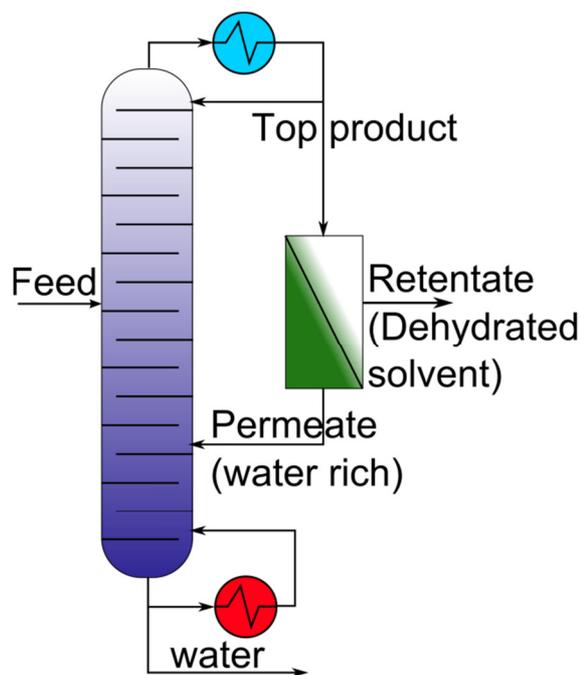


Figure 4: Distillation combined with pervaporation

Table 1: Results from dehydration run

Dehydration THF from 20 wt.% to 0.1 wt.%	Surface area	Average permeate water concentration
Only Pervaporation	50 [m <sup>2</sup> ]	82 wt.% (18 wt.% Loss of THF to permeate)
Distillation / Pervaporation	40 [m <sup>2</sup> ]	69 wt.% (no loss of THF)

A dehydration process based on pervaporation only requires only 25% more membrane surface in comparison to the hybrid system. This is due to the fact that dehydration at high water concentrations is much faster than at low water concentrations. The loss of THF to the permeate however amounts to 18 wt.%. In new installations the two options should be evaluated based on total cost of ownership calculations and environmental considerations.

### Energy and mass balances

In order to calculate the possible energy savings for a hybrid or membrane only system, mass and energy balances were made. The results are presented in table 2 and table 3. Table 2 shows the mass and energy balance for a conventional distillation with a reflux ration of 10 to achieve the desired water concentration in the top product. Table 3 holds the mass and energy balance for a hybrid system consisting of a distillation column with a reflux ratio of 2 and a membrane package to dehydrate the top product further to specifications. By reducing the reflux ratio and placing membranes after the top stream of the distillation column, the energy consumption of the system can be reduced by approximately 70 per cent. The energy consumption can even be reduced by approximately 80 per cent if the feed is directly dehydrated by means of pervaporation.

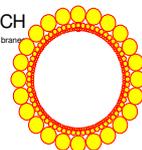


Table 2: Mass/Energy balance distillation only

Stream	$\Phi m$ [kg/h]	x_water [wt.%]	Q [kW]
Feed	1	456.25	0.2
Top	2	4017.51	0.0007
Reflux	3	3652.28	0.0007
THF	4	365.228	0.0007
Bottom	5	1001.24	0.9997
Reflux Bot	6	910.22	0.9997
Water	7	91.022	0.9997

Table 3: Mass/Energy balance combined distillation and pervaporation

Stream	$\Phi m$ [kg/h]	x_water [wt.%]	Q [kW]	Q_membranes only [kW]
Feed	1	456.25	0.2	114.42
Top	2	1180.29	0.05	175.29
Reflux top	3	786.86	0.05	
To mem	4	393.43	0.05	
Bottom	5	273.83	0.9993	
Reflux Bottom	6	182.55	0.9993	114.39
Water	7	91.28	0.9993	
Permeate	8	28.46	0.69	13.40
Retentate	9	364.97	0.0001	
<b>Energy reduction compared to distillation</b>			<b>72%</b>	<b>79%</b>

### Conclusion

In existing distillation processes a 72% energy reduction can be achieved by adding a pervaporation process. A side advantage of this solution is the capacity increase of the distillation column due to the reduction in load.

The full dehydration with membranes only requires the lowest CAPEX and energy cost but leaves a water rich waste stream with about 18 wt.% THF on the permeate side. This waste stream requires extra treatment or incineration.

The overall best option therefore is the combination of distillation with pervaporation to optimise energy consumption and eliminate waste streams since the permeate from the pervaporation process can be fed back to the distillation column.