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DEVELOPMENT OF A HYBRID SOLVENT RECOVERY PROCESS (COMBINATION OF DISTILLATION AND VAPOUR PERMEATION)

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INTRODUCTION

For the development of a new process for a specialty chemicals producer an adequate solvent recovery plant for the accumulated mixture of alcohols and water has to be designed. Different process alternatives will be compared based on the operation mode, recycling grade and process costs. Due to a secrecy agreement it is unfortunately not possible to talk about details of the customer process and about the different chemicals and solvents which are used in it. Therefore synonyms are used for the components.

PROCESS FUNDAMENTALS

Typical composition of the alcohol-water mixture:

Low boiling alcohol (LBA)	70 wt. %	Washing agent for the produced solid chemical
Water	17 wt. %	Washing agent for the produced solid chemical
High boiling Alcohol (HBA)	11 wt. %	Solvent for the synthesis
Medium boiler Alcohol (MBA)	0,7 wt. %	Side product of the synthesis

Specification for the recovered components

HBA $<$500 ppm impurities (water content!), purchase price app. 5–6 Euro/kg
LBA $<$5 wt. % impurities; amount app. 17 t per Batch
MBA Small amount of impurities to sell that as a side product or to minimize the disposal costs

Table 1. Boiling sequence in the system

Component	Comment
LBA	
MBA/Water	Homogeneous Low boiler azeotrope
MBA	
HBA/Wasser	Heterogeneous Low boiler azeotrope
HBA/Waterr/LBA	Heterogeneous Low boiler azeotrope
Water	
HBA	

START UP AND OPERATION EXPERIENCE OF THE HYBRID RECOVERY PLANT

The start up of the new recovery plant was in summer 2004. The main experiences with this plant since that are:

- The pH of the organic phase before entering the reboiler of the vapour permeation needs to be controlled (otherwise problems with material warranty). This set up will be done with an organic acid. At one of the first trials this organic acid was overdosed

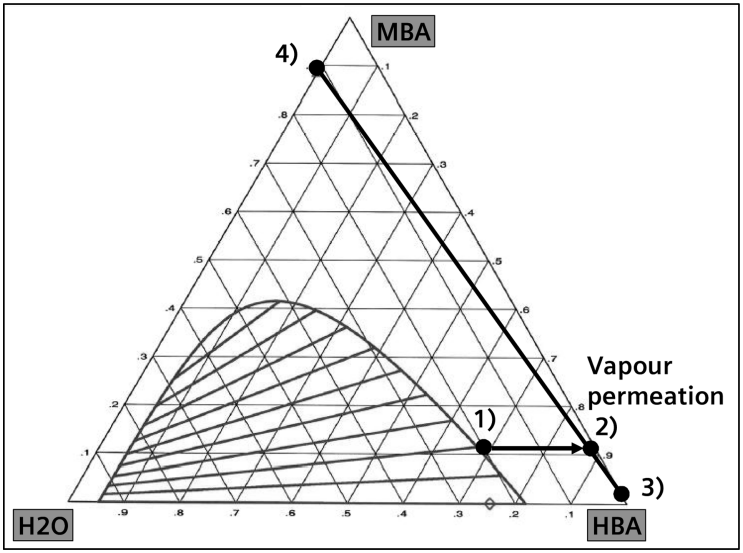
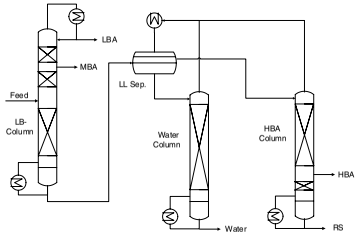
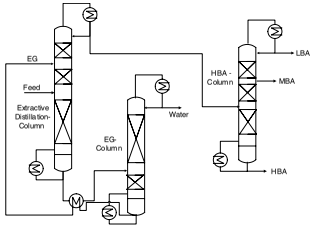
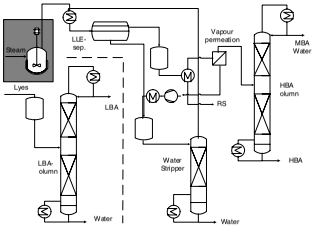


Figure 1. Ternary diagram H2O, HBA, MBA

Table 2. Comparison of the separation concepts

Separation concept	Conventional distillation	Extractive distillation (with Ethylenglykole as entrainer)	Hybrid process (as built)
Separation scheme			
HBA recovery rate	96%	93%	99%
Main advantages	The high recovery rate for HBA (>96%) The low energy consumption No necessity for an auxiliary component (e.g. entrainer) Easy heterogeneous distillation (five theoretical stages for the water stripper, and eight theoretical stages needed for the HBA stripper)	Easier separation than the conventional scheme (no vacuum needed) Less theoretical stages needed as for the conventional distillation (extractive distillation 24, EG column 10 and HBA column 14 theoretical stages) HBA is once distilled as top product (high boiler impurities are therefore separated) Well known process, only a small amount of experimental validation work was necessary	Easy distillation (HBA column 18, water stripper 5 and LBA column 10 theoretical stages) Highest recovery rate of all investigated concepts Lowest energy consumption of all investigated concepts
Main disadvantages	The high effort for the separation of the LBA (vacuum 400 mbara needed) HBA is a bottom product High experimental effort needed to validate the scheme (open questions LB column, influence of unknown impurities on the distillation and liquid–liquid separation)	Entrainer recovery necessary (elimination of high boilers which accumulate in the entrainer) High energy demand in comparison to the conventional scheme (factor 2, water is separated as a top product) Separation of MBA as a side draw leads to an increased loss of HBA, therefore the recovery rate of this process is smaller than that for the conventional one.	Additional process costs due to the fact that the membranes need to be replaced after some years. The process was calculated with a replacement of the membranes every two years. Due to low energy costs of that concept the hybrid process was still the cheapest option

and fed into the HBA column which leads to separation problems. Here a monitored pH adjustment solved the problem.

- Start up and especially shut down procedure for the membrane plant are more complicated than that for a simple distillation column. For the start up it is important build up a pressure (with nitrogen). Otherwise the displacement of the membrane material is possible. High water contents during start up are also problematic due to the mechanical stress of swelling of the polymere material depending on the water content. Therefore the start up of membrane plant is done with a high recycle stream of the retentate.
- After some trials with the HBA column the necessary stock up of MBA for the azeotropic distillation was fixed for the process.
- Up to today (March 2006, approx. 21 operating months) there is no performance loss of the membrane plant detectable.