Batch Extractive Distillation as a Hybrid Process: Separation of Azeotropes of Minimum Boiling Point

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For the separation of the two components (*A* and *B*) forming an azeotrope a special distillation method must be applied such as the extractive (ED) or heteroazeotropic distillation. In the case of ED a third, heavy component (solvent, *E*) is added to the mixture, which makes the separation of *A* and *B* possible without forming a new azeotrope. Batch distillation (BD) has always been an important part of seasonal, uncertain or low capacity and high-purity chemicals' production. The batch extractive distillation (BED) can simultaneously provide the advantages of BD and those of the ED. Recently the feasibility and performance of the BED were studied also for the non-conventional column configuration by several authors. Low and Sorensen (2002) optimising and comparing the performance of the batch rectifier and the Middle Vessel Column (MVC) for the BED of a minimum boiling point mixture have not obtained significantly better results for the MVC than for the much simpler batch rectifier.
The aim of this paper is to study the BED separation of minimum boiling point azeotropes by feasibility studies and rigorous simulation in the batch rectifier. The calculation results are presented for the mixtures acetone(*A*)-methanol(*B*) + water(*E*) and ethanol(*A*)-water(*B*) + ethylene glycol(*E*).
The BED process (Lang et al., 1994) consists of the following separation steps:

1. Start up under total reflux ($R=\infty$, $F=0$).
2. Purification of the distillate under $R=\infty$ and continuous feeding of *E* ($F>0$).
3. Production of *A* under continuous feeding of *E* ($0<R<\infty$, $F>0$).
4. Separation *B/E* ($0<R<\infty$, $F=0$).

The above process has among others has two important features, namely

1. The column is operated under significant reflux ratio ($R>0$) in Step 3.
2. The solvent can be fed into an inner plate of the column that can contain rectifying plates.

Düssel and Stichlmair (1995) and Stichlmair and Fair (1998) considered the BED as a hybrid process. They suggested the following steps for the separation of minimum azeotropes:

- Separation *A/B* by absorption ($R=0$, $F>0$).
- Separation *B/E* by distillation ($0<R<\infty$, $F=0$).

In order to study the hybrid process we extended the feasibility method of Lelkes et al. (1998) by making possible the variation of the heat condition of the solvent feeding (*q*) and that of the reflux for the BED. The results of the feasibility studies for the mixture ethanol-water + ethylene glycol show the decisive role of q (Fig. 1).

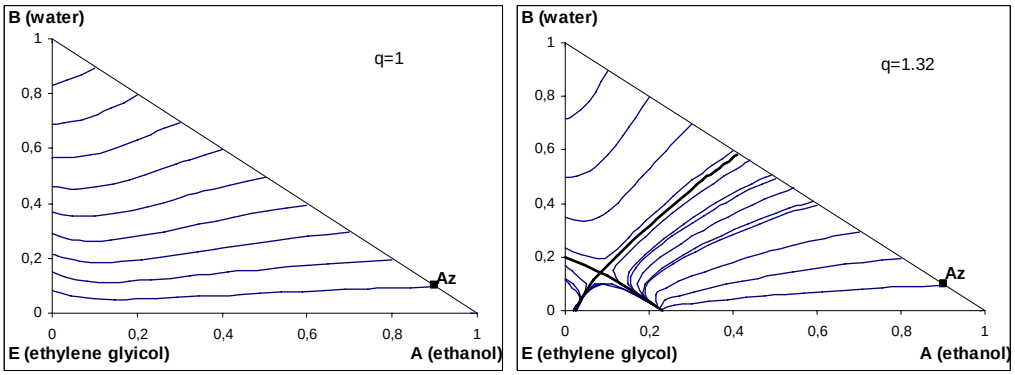

Fig. 1 The map of the extractive profiles
Solvent feeding: a. boiling point liquid ($q=1$) b. strongly subcooled liquid ($q=1.32$)

In the case q=1 the profiles arrive at the *BE* edge from the whole triangle, the component *A* can not be produced in the prescribed purity. In the other case a considerable part of the profiles arrive at the *AE* edge the separation can be feasible (with the aid of the feed plate since $\alpha_{A,E}$>>1).

For the more accurate modelling of the process rigorous simulation calculations were done by the CCBATCH and CCDCOLUMN professional simulators of Chemstations Inc. The hybrid process and the BED were compared for the mixture acetone-methanol +water whose BED separation was studied also by pilot plant experiments. By the BED we obtained the prescribed purity acetone with high recovery. By the hybrid process we were able to remove the methanol from the distillate only if we applied a very high feed flow rate (F). However the water content of the distillate was enormously high (Fig.2).

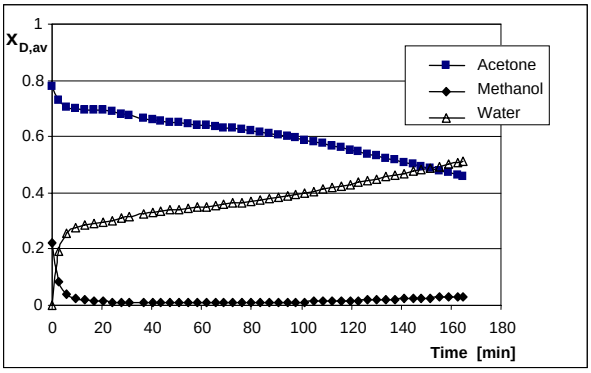

Fig. 2. The evolution of the top product composition (R=0, acetone-methanol+water)

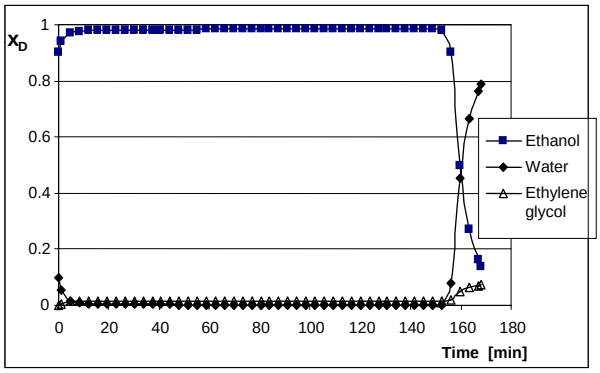

Fig. 3. The evolution of the distillate composition (R=0, ethanol-water+ethylene glycol)

Hence for producing a convenient acetone product the top product of the absorption step should be distilled again (an additional *A/E* separation step is indispensable).

The performance of the hybrid process was also studied for the mixture ethanol(*A*)-water(*B*) + ethylene glycol(*E*), where the difference between the boiling point of E and that of the original components is enormously very great. We could produce pure ethanol from azeotropic charge by applying moderate F (Fig. 3). However by BED even better results were achieved since we could considerably decrease the solvent consumption with a slight increase of the energy consumption of the *A* production step.

We concluded that by the hybrid process we can produce pure enough *A* in one step only if the *A/E* separation is very easy ($\alpha_{A,E}$>>1) and the difference between the boiling and freezing point of the solvent is very great (it can be subcooled to a great extent). The BED process provides higher degree of freedom, greater flexibility and can produce better results.

ACKNOWLEDGEMENT

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