SEPARATION OF TERNARY HETEROAZEOTROPIC MIXTURES IN A CLOSED MULTIVESSEL BATCH DISTILLATION-DECANTER HYBRID

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ABSTRACT

The feasibility of a novel batch distillation-decanter hybrid for simultaneous separation of ternary heterogeneous azeotropic mixtures is demonstrated through simulations. The multivessel column is operated without product withdrawal and the three components are accumulated in the vessels during one closed operation. Two ternary azeotropic mixtures of Serafimov's topological class 1.0-2 were studied. Simulated results for the system methanol/water/1-butanol verify the feasibility of the novel process in separating heterogeneous azeotropic mixtures. Results for the system acetone/water/toluene further justify the potential of the proposed distillation-decanter hybrid in separating close boiling temperature systems by the addition of a heavy heterogeneous entrainer.

INTRODUCTION

Much attention has been given lately to a special batch distillation column configuration named 'multi-effect batch distillation system' (Hasebe[1-3]) or 'batch distillation column with a middle vessel' (Davidyan[4], Barolo[5-6], Warter[7-8]) or 'middle vessel column' (Cheong[9-11], Rodriguez-Donis[12]) or 'multivessel batch distillation column' (Wittgens[13], Skogestad[14], Hilmen[15-16]).

The novel column configuration was first mentioned by Robinson and Gilliland[17] in 1950, but the use of such a column for the separation of binary and multicomponent mixtures was not analysed until 1970, independently by Bortolini and Guarise[18] and Treybal[19]. The interest into this column configuration was renewed after the work of Hasebe et al.[1] and since then the complex batch column often appears in the related literature.

The multi-vessel column (MVC) is a combination of a batch rectifier and a batch stripper. The column has both a rectifying and a stripping section and therefore it is possible to obtain a light and a heavy fraction simultaneously from the top and the

bottom of the column while an intermediate fraction may also be recovered in the middle vessel. Several modifications of the MVC have been suggested in the literature. One with a liquid bypass, where the liquid stream from the rectifying section bypasses the middle vessel and enters the stripping section, one where both liquid and vapour streams from the top section enter the middle vessel and one with a vapour bypass from the bottom section to the top section of the column. In our work we use the vapour bypass modification, which is the most common in the literature, as shown in Figure 1a.
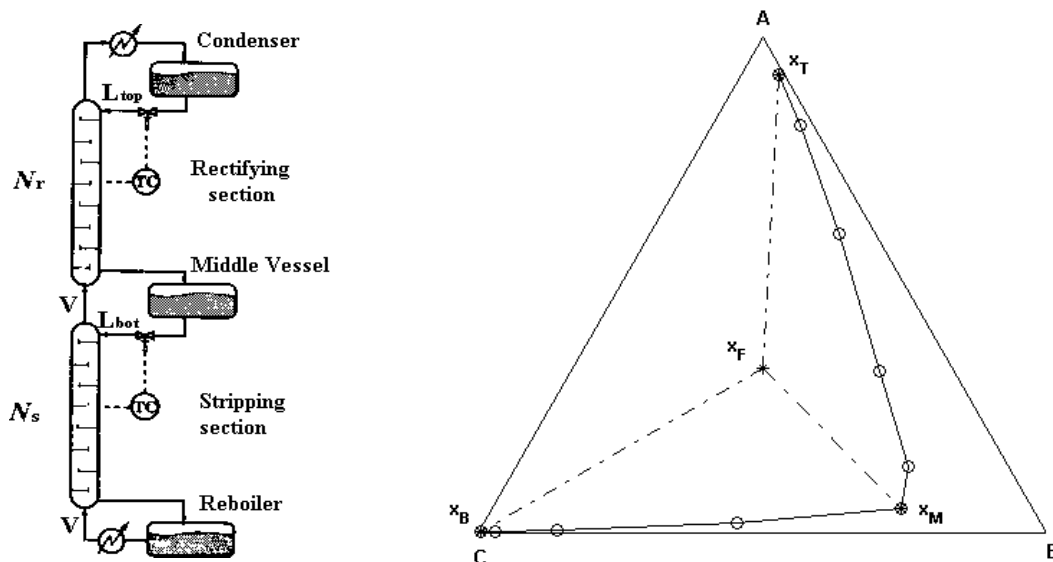

Figure 1: a) Multivessel batch column (MVC). b) Steady state column profile (solid line with open circles) and material balance triangle (dashed lines)

Until recently most of the work on the MVC was for ideal or constant relative volatility systems. Hasebe et al.[1-3] studied some characteristics of the column and presented simulation results for binary and ternary mixtures. They also proposed the total reflux (closed) operation of such a column. Davidyan et al.[4] presented a rigorous mathematical analysis of the MVC and tried to describe the dynamic behaviour of the column. Meski and Morari[20] provided a limiting analysis of a mathematical model for the MVC under the assumptions of negligible holdup on the trays and constant molar flows. Barolo et al.[5-6] performed experiments in a continuous column modified in order to resemble the MVC. They implemented different control configurations and also discussed the effect of some operating parameters on column performance. For the closed operation of the MVC, a feedback control structure, based on temperature controllers, has been proposed by Skogestad et al.[14]. The feasibility of this simple control strategy was demonstrated both by simulations and experiments by Wittgens et al.[13]. A constant relative volatility quaternary system was studied and at the end of the process four pure products were accumulated at the vessels.

The first who tried to study the performance of the MVC in the separation of azeotropic mixtures was Safrit et al.[21-22]. They studied the case of extractive batch distillation in the MVC, with a heavy entrainer added continuously at the top of the column. Warter et al.[7] and Hilmen et al.[15] also studied the performance of extractive batch distillation in the MVC and reported some advantages of the novel process over conventional batch extractive distillation. In a series of three papers Cheong et

al.[9-11] developed a mathematical model in order to study the qualitative dynamics of the MVC when it is used to separate multicomponent azeotropic mixtures. The theoretical insights from the analytical tools developed, were also verified by simulations. Warter et al.[8] has showed simulated results for the separation of binary azeotropic mixtures by using homogeneous entrainers. They showed ways to cross the distillation boundaries by placing the feed at the concave side of the boundary and by manipulating the vapour flows in the two sections of the column. In that way all three original components can be recovered. Hilmen[16] provided also simulated results on the closed multivessel batch distillation of ternary homogeneous azeotropic mixtures when the control strategy proposed by Skogestad et al.[14] was implemented. In a recent work Rodriguez-Donis et al.[12] provided simulated results when heterogeneous entrainers were used instead. In this case a decanter should be combined with the MVC for doing the liquid-liquid split (distillation-decanter hybrid process).

The present work addresses heterogeneous azeotropic batch distillation in a distillation-decanter hybrid. The basic principle behind the novel process is that the heteroazeotrope achieved in the decanter vessel of the MVC is separated into its two liquid phases by cooling and decantation. One phase is then recycled to the column while the other is accumulated in the decanter vessel. In this way it is possible to cross the distillation boundaries by decantation and the ternary heteroazeotropic mixture is separated into three pure components in one distillation-decanter hybrid column.

The MVC is operated under total reflux or better as a closed system since total reflux is only achieved as we approach steady state. The main advantage of the closed MVC is the simplicity of operation, since no product change-overs are required but rather pure products are accumulated in the vessels at the end of the process. Additionally, the 'total reflux' operation utilises the maximum attainable separation in the column and is especially advantageous for high purity separations. Finally, this operating mode allows us to make direct use of the distillation lines or residue curves, thus extracting valuable information about the possible products in the vessels as we reach steady state.

The correspondence between the distillation lines and the steady state column profile is simple and straightforward. 'The steady state vessel product compositions of a closed multivessel batch distillation column are connected by a distillation line with the given number of equilibrium trays. In addition the material balances must be satisfied' (Hilmen[16]). At steady state the material balances for the closed MVC can be written as:

$$M_F = M_T + M_M + M_B$$

$$M_F \, \mathbf{x}_F = M_T \, \mathbf{x}_T + M_M \, \mathbf{x}_M + M_B \, \mathbf{x}_B \Rightarrow$$

$$M_M \, (\mathbf{x}_F - \mathbf{x}_M) = M_T \, (\mathbf{x}_T - \mathbf{x}_F) + M_B \, (\mathbf{x}_B - \mathbf{x}_F)$$

The above material balances reveal that the vessel holdups must obey the so-called lever rule. In addition the vessel compositions must lie on the same distillation line because total reflux is reached at steady state. As also noted by Hilmen[16] 'the

feasible region of vessel compositions in the MVC are enclosed by the feed distillation line and the borders of the current feed distillation region and by the material balance triangle connecting the unstable node, the stable node and one of the saddles of the current feed distillation region', as shown in Figure 1b.

MODEL

The *dynamic* model for the middle vessel column used in our simulations consists of:
- Overall and component mass balances
- Vapour-liquid equilibria[23] modeled by UNIQUAC equation and liquid-liquid equilibria[24-25] based on experimental data
- Indirect level control in the vessels with P temperature controllers, as proposed by Skogestad[11], for step 1 of the process. Direct level control in the decanter with a PI level controller for step 2 of the process

The model is based on the following assumptions:
- Staged distillation column sections (N_r and N_s stages in the rectifying and stripping section, respectively)
- Equal constant molar vapour flows in both sections of the column (simplified energy balances)
- Constant molar liquid holdup on all stages, negligible compared to the holdup on the vessels (neglecting liquid dynamics)
- Negligible vapour holdup
- Perfect mixing and equilibrium in all stages
- Ideal vapour phase
- Constant pressure at P = 1.013 bar

All simulations were performed in Matlab[26] by using the integration routine ODE15s.

RESULTS

Methanol – Water – 1-Butanol   (System 1)
The first mixture that has been studied is Methanol – Water – 1-Butanol. Water and 1-butanol exhibit a heterogeneous azeotrope and an immiscibility gap over a limited region of ternary compositions exists (Figure 2a). The stability of the stationary points of the system is also shown in this figure. The system belongs to Serafimov's topological class 1.0-2 (Hilmen[27]). One distillation boundary, running from methanol (unstable node) to the binary heteroazeotrope (saddle), divides the composition space in two regions, thus limiting the feasible products under distillation. For example, a feed F placed at the left feed region will provide the unstable node (methanol) as a top product, the saddle (binary heteroazeotrope) as a middle vessel product and the stable node (1-butanol) as a bottom product, provided that we have enough stages in each column section.
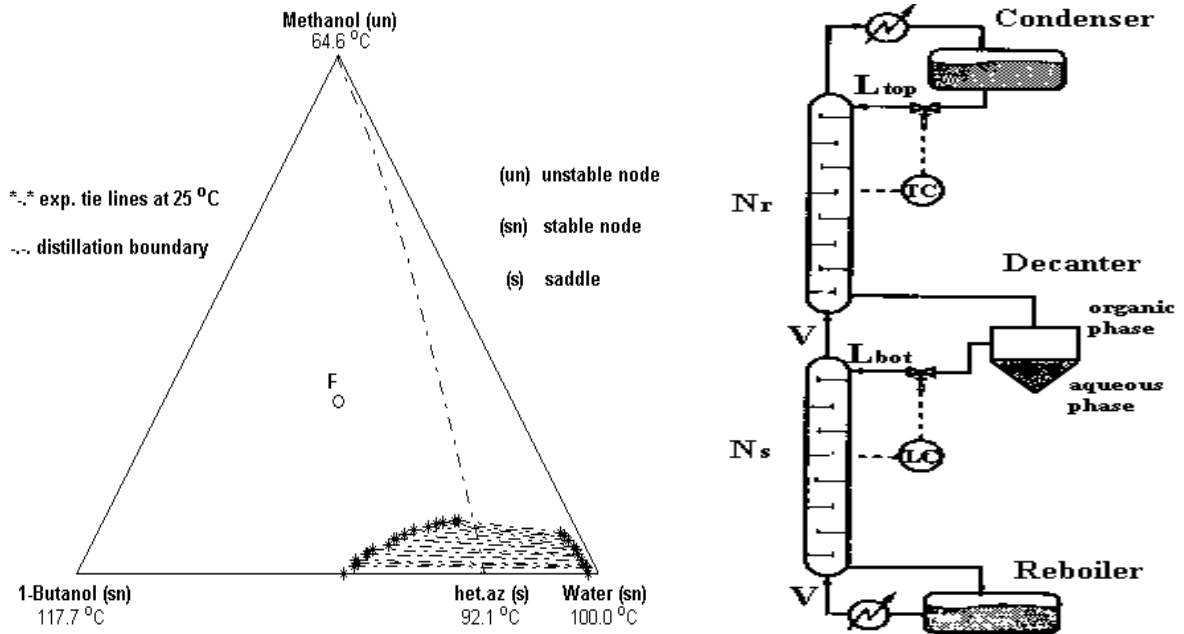
Figure 2: a) Azeotropic mixture of Serafimov's topological class 1.0-2.
b) Multivessel batch distillation – decanter hybrid

Water is impossible to be taken as a distillation product in this column since it belongs to a different distillation region and the distillation boundary cannot be crossed under total reflux. Therefore not all three original components can be separated simultaneously in one closed operation of the multivessel column. That is, for example, the case for homogeneous azeotropic mixtures as mentioned by Hilmen[16]. However, the fact that the azeotrope accumulated in the middle vessel is heterogeneous provides a straightforward way of overcoming the azeotropic composition and cross the distillation boundary, namely by decantation. In this way the column can be operated in both distillation regions and all three components can be recovered in the vessels in one closed operation. Thus, a distillation-decanter hybrid seems like a promising alternative.

In the following we will show that the multivessel batch column with a decanter taking the place of the middle vessel provides a feasible way of separating heterogeneous azeotropic mixtures. The proposed novel process can be seen in Figure 2b. The closed operation of such a hybrid column certainly simplifies the process since 'the column runs by itself' and exhibits advantages over other operating techniques as, for example, the 'steering the middle vessel' technique proposed by Cheong et al.[9-11] and implemented for heterogeneous azeotropes by Rodriquez-Donis[12].

The operating procedure followed in our simulations is:
Step 1. *Start up:* Built up the composition profile at the column. No decantation is performed during this step.
Step 2. *Decantation:* Decantation starting. One of the immiscible phases is refluxed back in the column. Final products are accumulated in the vessels at the end of this step.

Our simulations for system 1 showed the following patterns
Step 1. The feed (x_F) is placed at the left feed region. The composition profile in the column is built up. The final products in the vessels can be easily predicted by

checking the stability of the singular points at this feed region. Since methanol is the unstable node of the region, it appears in the top vessel. The saddle heteroazeotrope is accumulated in the middle vessel while 1-butanol appears in the bottom vessel, as the stable node of the region. Figure 3a illustrates Step 1 of the process and shows the evolution of the compositions in all vessels. The steady state liquid profile in the column and the final products in the vessels (x_T, x_M, x_B) are also depicted in the figure.
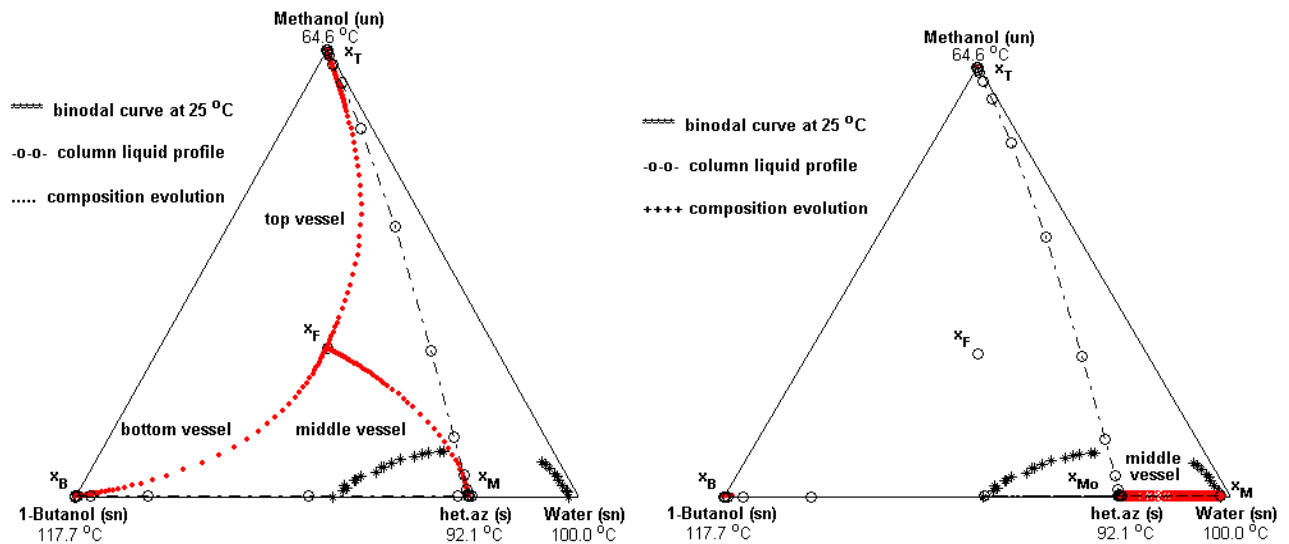

Figure 3: Separation of the system Methanol – Water – 1-Butanol. a) Step1 b) Step 2

Step 2. The heteroazeotrope accumulated in the middle (decanter) vessel consists of two immiscible liquid phases, one rich in water (aqueous phase) and one rich in 1-butanol (organic phase). If we decant the two phases and reflux the organic phase back in the column, the 1-butanol previously 'trapped' in the azeotrope is stripped down in the column and is accumulated in the bottom vessel. At the same time, the aqueous phase is accumulated in the middle vessel while the methanol stays at the top of the column. So all three original components can be recovered in the vessels at the end of the process.

Our simulations show that this is the case. Figure 3b exhibits how the middle vessel is getting enriched in water. The water composition in the middle vessel starts at the heteroazeotropic point (x_{Mo}=0.782) and ends up at the composition of the aqueous phase determined by the liquid-liquid experimental data (x_M=0.98). The 1-butanol, contained in the heteroazeotrope, is now refluxed back in the stripping section of the column and enters the bottom vessel. At the end pure methanol is collected in the top vessel ($x_{methanol}$≈1), water in the middle vessel (x_{Water}=0.98) and pure 1-butanol in the bottom ($x_{1-Butanol}$≈1). The corresponding recoveries are $r_{Methanol}$≈100%, r_{Water}≈100%, $r_{1-Butanol}$=95.8%. All simulation data, initial conditions and steady state results are presented at the Appendix.

Acetone – Water – Toluene (System 2)
System 1 is rather a literature example and it was chosen in order to verify the feasibility of the proposed multivessel-decanter hybrid in separating heteroazeotropic mixtures. The decanter performs the wanted liquid-liquid split of the heteroazeotrope,

while the stripping section further purifies one of the components forming the azeotrope. However, this separation can be also performed in a common batch rectifier-decanter hybrid and the rectifying section of the column performs a separation (methanol-water) which is of not particular interest and difficulty. We will show now that the proposed process provides further advantages compared to a batch rectifier-decanter hybrid by presenting an example where the rectifying section is performing a more interesting and difficult separation.

One such example is the system Acetone – Water – Toluene. Acetone with high purity is difficult to be recovered from its aqueous solutions since the binary system exhibits a tangent pinch at molar fractions of acetone greater than 0.95. Such close boiling temperature systems are of great industrial interest since high-purity separation by ordinary distillation becomes uneconomical because high number of trays and high reflux ratios are necessary.

The common practice is to add a third component (entrainer) in the system, which acts preferentially with one of the original components and eases the separation. Toluene can be used as such a suitable entrainer in our case. Toluene is heavier than both acetone and water forms a heterogeneous azeotrope with water. The mixture exhibits an immiscibility gap over a wide range of ternary compositions. There is one distillation boundary running from acetone to the heteroazeotrope as can be seen in figure 4. The system belongs to Serafimov's class 1.0-2 like system 1.

The procedure followed in our simulations was the same as for system 1.
Step 1. The initial feed (x_F) was placed at the left feed region where acetone is the unstable node (top vessel), toluene is the stable node (bottom vessel) and the heteroazeotrope is a saddle and therefore it is accumulated in the middle vessel. The stripping section performs the separation water-toluene and the binary azeotropic composition is reached in the middle vessel. The rectifying section is devoted to the binary separation acetone-water and therefore the steady state liquid profile in the top section follows the distillation boundary (Figure 5a). The distillation boundary touches asymptotically the acetone-water vertex at high acetone concentrations, which verifies the difficulty of the separation (tangent pinch) in the top of the column. Therefore more stages are required in the rectifying section of the column than in the stripping section.
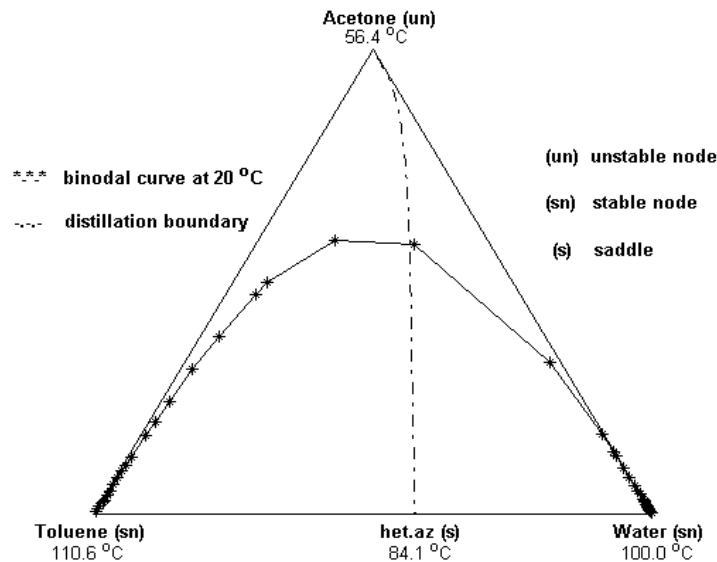
Figure 4: The system Acetone – Water –Toluene (Serafimov's class 1.0-2)

Step 2. After building up the composition profile in the column we start decanting the two immiscible liquid phases in the middle vessel. The entrainer rich (organic) phase, containing a very small amount of water (see experimental binodal curve), is refluxed back in the column. The aqueous phase, which is almost pure water, is accumulated in the middle vessel (Figure 5b). At the end of the process the aqueous phase is collected in the middle vessel (x_M=0.995). The top vessel contains acetone (x_T=0.988) and the entrainer (toluene) is collected in the bottom vessel (x_B≈1). The corresponding recoveries are $r_{Acetone}$=100%, r_{Water}=86%, $r_{Toluene}$=97.3%. Detailed steady state results are given in the Appendix.
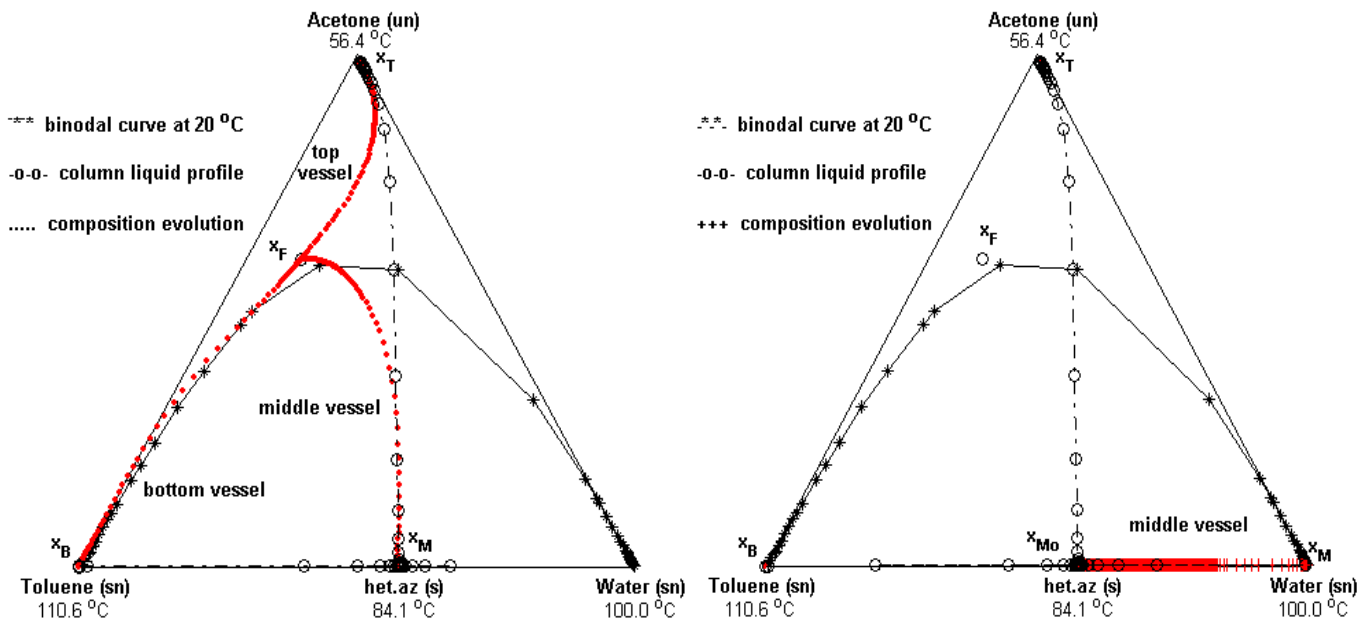

Figure 5: Separation of the system Acetone – Water – Toluene. a) Step 1 b) Step 2

FUTURE WORK

More heterogeneous azeotropic ternary systems will be investigated in the multivessel distillation-decanter hybrid. The following systems exhibiting one heteroazeotrope will be a subject of future research.

Ethyl acetate – Water – Acetic Acid (Serafimov's class 1.0-1a)

Acetic acid and water form a tangent pinch and high purity acetic acid is difficult to be recovered from its aqueous solutions. The addition of a light heterogeneous entrainer eases the separation. One such entrainer can be ethyl acetate. The corresponding distillation lines map modeled by UNIQUAC along with the stability of the stationary points is shown in figure 6a. We ignore for the moment the heterogeneous composition region and the peculiarities the distillation lines exhibit inside this region. There is no distillation boundary and the binary heteroazeotrope between water and ethyl acetate is the unstable node of the system. Thus, it will appear in the top of the column. The bottom product will be acetic acid (stable node). Depending on the feed composition the middle vessel product can be either ethyl acetate or water (saddles). If we place the feed at the right bottom feed region then pure water accumulates in the middle vessel. The heteroazeotrope in the top vessel is decanted and the aqueous phase is refluxed back in the column. Thus the top vessel is steadily enriched in ethyl acetate (until the point determined by the liquid-liquid equilibrium), while the middle vessel is enriched in water. Acetic acid remains in the bottom of the column because of low volatility. At the end of the process three pure products are accumulated in the vessels.

Water – Acetic Acid – Butyl Acetate (Serafimov's class 1.0-1b)

In this case a heavy heterogeneous entrainer (butyl acetate) is added in the original binary mixture of water-acetic acid. The corresponding ternary phase diagram is shown in figure 6b. There exists again no distillation boundary. Water forms a heterogeneous azeotrope with butyl acetate. This heteroazeotrope is the unstable node of the system and appears in the top of the column. Butyl acetate is accumulated in the bottom and with the right placement of the feed acetic acid is possible to be collected in the middle vessel. A decanter in the top 'breaks' the heteroazeotrope and the entrainer rich phase is refluxed back in the column. Thus, the butyl acetate previously 'trapped' in the top vessel is now running down the column and enters the bottom vessel. At the end of the process the aqueous phase is collected in the top, acetic acid in the middle vessel and the entrainer in the bottom vessel.
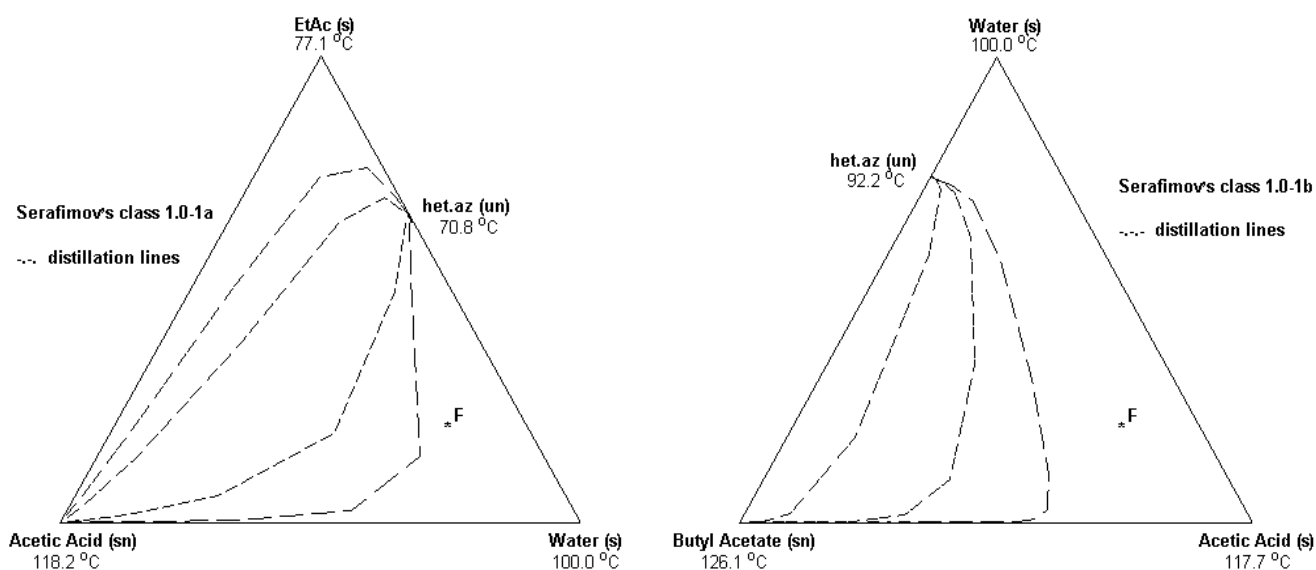
Figure 6: a) The system Ethyl Acetate – Water – Acetic Acid
b) The system Water – Acetic Acid – Butyl Acetate

CONCLUSIONS

Two ternary heterogeneous azeotropic systems, both classified at Serafimov's topological class 1.0-2, were presented in this work. The feasibility of their separation in the closed multivessel batch distillation-decanter hybrid was investigated by simulations in Matlab.

The results for the mixture methanol-water-1-butanol (system1) proved the feasibility of the proposed novel process. The heteoazeotrope formed by water and 1-butanol was 'broken' by decantation, while distillation was used in order to achieve further purification of the original components. At the end of the process three pure components were accumulated in the vessels.

Results for the system acetone-water-toluene further justified the potential of this novel hybrid process. The column was used for the separation of a close-boiling temperature (tangent pinch) acetone-water mixture by using a heavy heterogeneous entrainer (toluene) and three pure products were accumulated in the vessels at the end of the process.

Azeotropic systems classified at Serafimov's topological class 1.0-2 can be separated in the novel column simultaneously in one closed operation. Future work will investigate the possibility of separating other classes of azeotropic mixtures, like for example systems belonging to Serafimov's topological class 1.0-1a and 1.0-1b.

NOTATION

M   Liquid holdup (kmol)
x   Liquid compositions
r   Recoveries
1,2   Process steps

Subscripts

F   Feed
T   Top vessel
M   Middle vessel
B   Bottom vessel
r   Rectifying section
s   Stripping section

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APPENDIX

Table 1: Simulation data and initial conditions

System 1: Methanol – Water – 1-Butanol	
Feed composition	$\mathbf{x}_{F0}$ = [1/3, 1/3, 1/3]
No. of trays per section	N_r = 25, N_s = 15
Initial charge	M_F = 5.385 kmol
Initial condenser holdup	M_{T0} = 0.035 kmol
Initial middle vessel holdup	M_{F0} = 5.000 kmol
Initial reboiler holdup	M_{B0} = 0.250 Kmol
Trays holdup (constant)	M_i = 1/300 kmol
Vapor flow (constant)	V = 5 kmol/h

System 2: Acetone – Water – Toluene	
Feed composition	$\mathbf{x}_{F0}$ = [0.6, 0.1, 0.3]
No. of trays per section	N_r = 65, N_s = 25
Initial charge	M_F = 5.385 kmol
Initial condenser holdup	M_{T0} = 0.035 kmol
Initial middle vessel holdup	M_{F0} = 5.000 kmol
Initial reboiler holdup	M_{B0} = 0.250 Kmol
Trays holdup (constant)	M_i = 1/300 kmol
Vapor flow (constant)	V = 5 kmol/h

Table 2: Simulation results

System 1: Methanol – Water – 1-Butanol

Step 1	**Condenser**	**Middle Vessel**	**Reboiler**
Holdup (kmol)	1.778	2.240	1.267
$X_{Methanol}$	**1.000**	0.000	0.000
X_{Water}	0.000	**0.782**	0.000
$X_{1\text{-}Butanol}$	0.000	**0.218**	**1.000**
Recovery (%)	-	het.az	-

Step 2			
Holdup (kmol)	1.778	1.819	1.688
$X_{Methanol}$	**1.000**	0.000	0.000
X_{Water}	0.000	**0.980**	0.000
$X_{1\text{-}Butanol}$	0.000	0.020	**1.000**
Recovery (%)	100	100	95.8

System 2: Acetone – Water – Toluene

Step 1	**Condenser**	**Middle Vessel**	**Reboiler**
Holdup (kmol)	3.285	0.750	1.250
$X_{Acetone}$	**0.988**	0.000	0.000
X_{Water}	0.012	**0.575**	0.000
$X_{Toluene}$	0.000	**0.425**	**1.000**
Recovery (%)		het.az	

Step 2			
Holdup (kmol)	3.285	0.457	1.543
$X_{Acetone}$	**0.988**	0.000	0.000
X_{Water}	0.012	**0.995**	0.000
$X_{Toluene}$	0.000	0.005	**1.000**
Recovery (%)	100	86.0	97.3

Keywords: Batch distillation
Multivessel column
Heterogeneous azeotropic mixtures
Hybrid processes
Distillation-decanter hybrid