R&D NOTES

Plantwide Control of an Isopropyl Alcohol Dehydration Process

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Introduction

Solvents are widely used in many industries. The need to recover the solvent requires the subsequent separation of the solvent from components that have been produced during reactions. One important example is an organic solvent that must be separated from water. The separation is frequently made difficult by the occurrence of complex vapor-liquid equilibrium that generates azeotropes.

Sommer and Melin[1] recently studied the steady-state economics of several alternative processes for the dehydration of liquid organic solvents. They explored both conventional distillation processes and hybrid processes that combine distillation with membranes. Their objective is to use the strengths of membrane separation to complement the weaknesses of distillation. Dynamics and control are not considered in their article.

The current industrial standard for the dehydration of IPA is the use of extractive distillation. Sommer and Melin[1] provide a fairly detailed description of this process, and their design specifications and parameter values are used in this paper to set equipment sizes and operating conditions.

The control of extractive distillation is discussed by Grassi.[2] Temperature control in the extractive column and the solvent recovery column are recommended, as well as ratioing the flow rate of the extractive solvent to the feed flow rate. This basic structure is used in this article, but a more effective way to manage the solvent-to-feed ratio is proposed. Anderson et al.[3] discuss the interaction between process design and dynamic controllability of extractive distillation systems. Zheng et al.[4] explore an extractive distillation column that exhibits input multiplicity.

The commercial process simulation programs Aspen Plus and Aspen Dynamics are used in the work. The Unifac physical property package is used.

Process Studied

The feedstream is a binary mixture of 2-propanol (isopropyl alcohol, IPA) and water. This nonideal mixture has an azeotrope that makes it impossible to obtain complete separation in a single column. The composition of the minimum-boiling homogeneous azeotrope at a pressure of 1 atm is 87.6 wt % IPA, with a temperature of 353.4 K. The normal boiling point of IPA is 355.4 K. Figure 1 gives the Txy diagrams at 1 atm and at 10 atm, which show that there is very little pressure dependence. This precludes the use of pressure-swing azeotropic distillation as an effective way to separate the components. Chien et al.[5] study the use of a light entrainer (cyclohexane) to dehydrate IPA through the formation of a heterogeneous azeotrope, which produces a process with a column and a decanter.

The conventional process presented by Sommer and Melin (2004) uses extractive distillation with a heavy entrainer. The flowsheet features three distillation columns, as shown in Figure 2. The feed is 1875 kg/h of an 80 wt % IPA and 20 wt % water binary mixture at 350 K. It is fed on Stage 10 of a 19 stage (17 trays) column C1 operating at 1.1 atm condenser pressure, giving top and bottom temperatures of 356 and 379 K, respectively. The reflux ratio is 0.637. The bottoms product is 99.99 wt % water. The distillate is 86.14 wt % IPA, which is close to the azeotropic composition. Reboiler heat input is 716 kW.

The distillate is fed to Stage 30 (using Aspen notation of numbering from the condenser down) of the 60-stage second column C2. An extractive solvent of ethylene glycol is fed on Stage 7 at a flow rate of 5,000 kg/h, and a temperature of 373 K. The specifications for this column are a distillate product that is 99.91 wt % IPA, and a bottoms stream with ppm concentrations of IPA. The column pressure is 0.8 atm, giving top and bottom temperatures of 350 and 437 K, respectively. The reboiler heat input is 816 kW.

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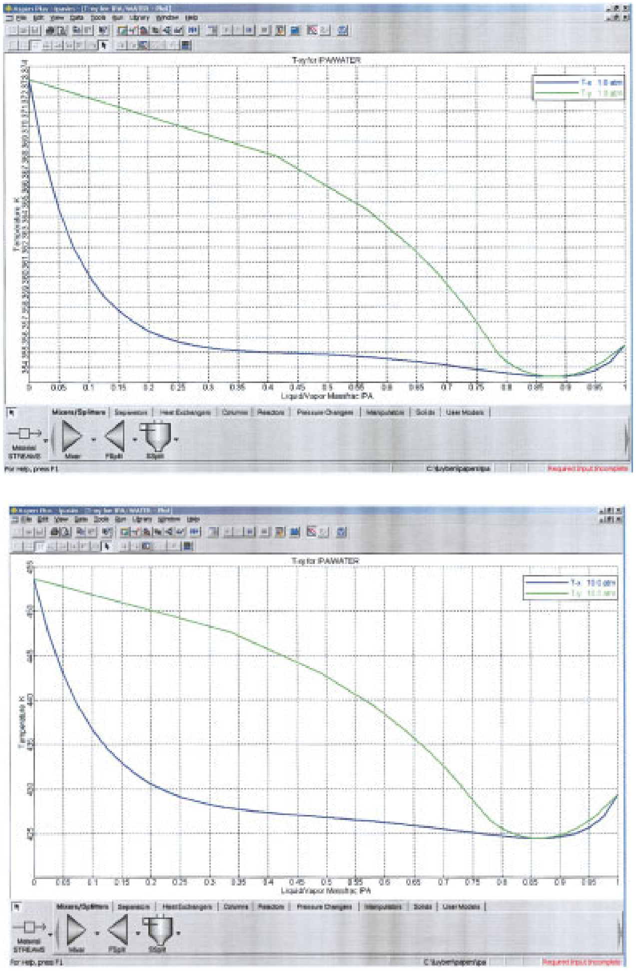

Figure 1. (A) IPA/Water at 1 atm. (B) IPA/Water at 10 atm.

[Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Sommer and Melin[1] specify a reflux ratio of 0.5 for this column. However, our simulations show that a reflux ratio of 1 is required to attain the separation. This is discussed in more detail in the next section.

The bottoms of C2 are fed on Stage 12 of the third column C3, which has 25 stages and operates at 0.4 atm, giving top and bottom temperatures of 349 and 447 K, respectively. The distillate product is 99.98 wt % water. The bottoms stream is essentially pure ethylene glycol, which is cooled and recycled back to C2. The reboiler heat input is 410 kW, and the reflux ratio is 1.5.

Table 1 gives details of the design parameters and operating conditions at steady state.

Table 1. Design Parameters for Three Columns

		C1	C2	C3
Reflux Ratio		0.637	1.0	1.5
Flows (kg/h)	D	1742	1501	241
	B	133	5241	5000
	F	1875		
	Solvent		5000	
Compositions				
(wt % IPA)	Feed	80		
(wt % IPA)	Distillate	86.1	99.91	0.02
(wt % H_2O)	Bottoms	99.99	4.59	
Pressure (atm)		1.1	0.8	0.4
Temperatures (K)	Reflux Drum	356	350	349
	Base	379	437	447
Heat Duty (kW)	Reboiler	716	816	410
Diameter (m)		0.584	0.609	0.552
Total stages		19	60	25

Temperature Profiles and Separation Results

Temperature profiles for the three columns are shown in Figure 3. Columns C1 and C3 have fairly sharp temperature breaks, which indicate that these columns can probably be fairly effectively controlled by holding an appropriate tray temperature constant. Stage 17 in C1 is selected, and is controlled at 368 K. Stage 4 in C3 is selected, and is controlled at 390 K.

Column C2 is more of a problem. The shape of the temperature profile has several breaks and plateaus, and it is not obvious if temperature control will be effective nor what tray to select.

A series of steady-state runs were made to study the effect of various parameters on the separation attained in the extractive distillation column C2. The "design spec/vary" capability of Aspen Plus was used to hold the purity of the distillate product at 99.91 wt % IPA by varying the flow rate of the distillate.

There are two other degrees of freedom in this column:

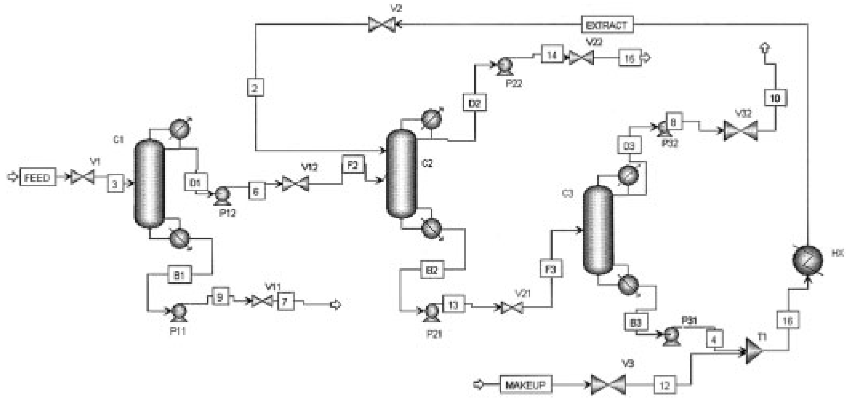

Figure 2. Steady-state aspen plus flowsheet.

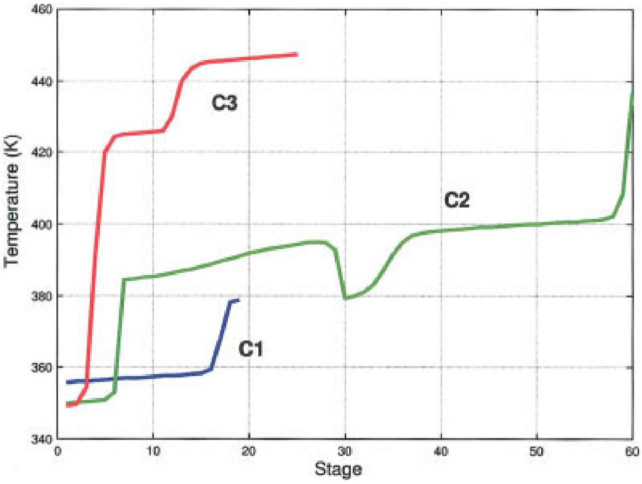

Figure 3. Temperature profiles.

[Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

reflux ratio and solvent flow rate. Figure 4 shows how the temperature profile is affected by changes in these two parameters. In the top graph the reflux ratio is constant with a value of 1. The flow rate of the extractive solvent is varied. As flow rate is decreased, the temperature break in the lower part of the column (Stages 30 to 60) moves towards the bottom of the column (to the right in the top graph). The effect of this is to drop more IPA out the bottom of the column. The IPA concentration must be kept very small to avoid loss of product and contamination of the water produced in the downstream column C3.

The concentration of IPA in the bottoms is 1×10^{-14} wt % IPA at the design conditions of 5,000 kg/h of solvent. When solvent is reduced to 4,500 kg/h, the bottoms composition is 2×10^{-12} wt % IPA. For flow rates below this value, the composition is extremely sensitive to solvent flow rate. For example, at 4,400 kg/h the bottoms composition increases dramatically to 0.6 wt % IPA. Figure 5 shows this extreme sensitivity.

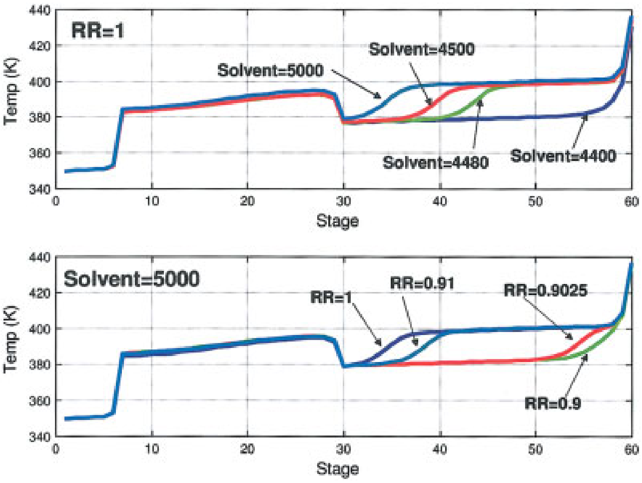

Figure 4. Effect of RR and solvent flow on temperature profiles in C2.

[Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

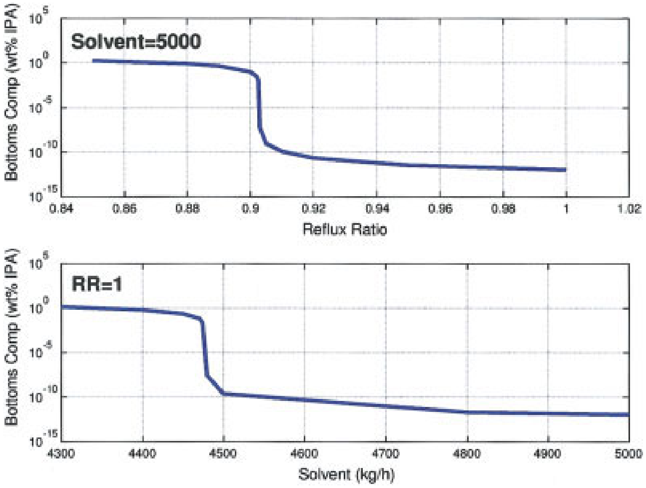

Figure 5. Effect of RR and solvent flow on bottoms composition.

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The lower graph in Figure 4 shows how changing the reflux ratio affects the column temperature profile with the solvent flow rate fixed at 5,000 kg/h. Reducing the reflux ratio shifts the profile towards the base of the column and raises IPA impurity in the bottoms.

These results suggest that a control structure that holds a constant temperature of 395 K on Stage 50, and maintains a constant reflux ratio might provide effective control of C2.

Basic Control Structure

Figure 6 gives the initial plantwide control structure using the recommendations given by Grassi.[2] The column tray temperature controllers have 1-min deadtimes, and are tuned by performing a relay-feedback test and using the Tyreus-Luyben settings. Temperature transmitter ranges are 300 to 500 K. All level controllers are proportional with gains of 2. Liquid holdups are sized to provide 5 min of holdup time when 50% full, except for the base of column C3, which is sized to provide 10 min.

Column C1:

1. Feed is flow controlled.
2. Stage 17 temperature is controlled at 368 K by reboiler heat input ($K_C = 0.82$ and $\tau_I = 10.6$ min).
3. Pressure is controlled by condenser heat removal.
4. Reflux ratio is held constant.
5. Reflux-drum level is controlled by distillate flow.
6. Base level is controlled by bottoms flow.

Column C2:

1. The extract solvent flow is flow controlled.
2. The temperature of the solvent is controlled at 373 K by manipulating the heat removal in the cooler (HX).
3. Stage 50 temperature is controlled at 395 K by reboiler heat input ($K_C = 2.1$ and $\tau_I = 10.6$ min).
4. Pressure is controlled by condenser heat removal.
5. Reflux ratio is held constant

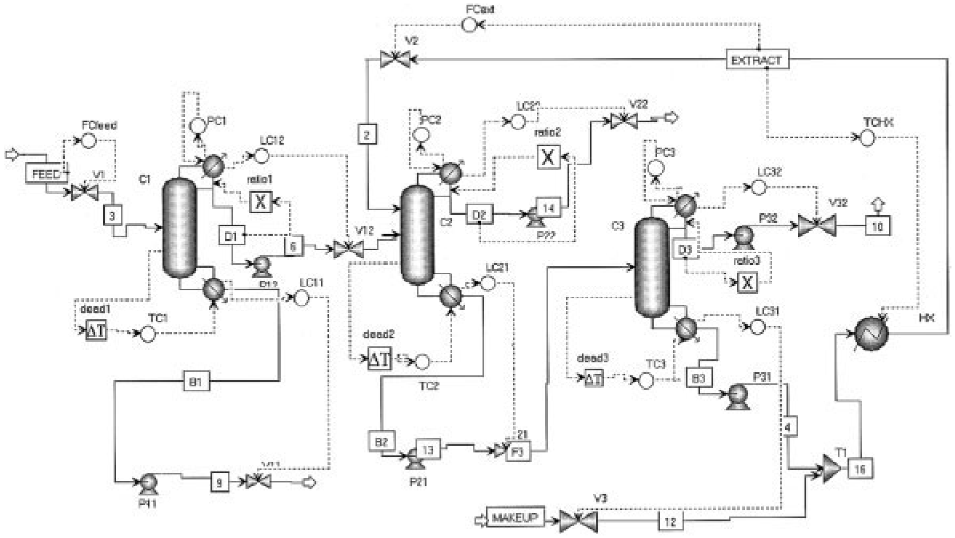

Figure 6. Plantwide control structure.

6. Reflux-drum level is controlled by distillate flow.
7. Base level is controlled by bottoms flow.

Column C3:

1. Stage 4 temperature is controlled at 390 K by reboiler heat input ($K_C = 0.41$ and $\tau_I = 11.2$ min).
2. Pressure is controlled by condenser heat removal.
3. Reflux ratio is held constant.
4. Reflux-drum level is controlled by distillate flow.
5. Base level is controlled by the flow rate of the makeup ethylene glycol. The losses of the solvent are very small, so this level essentially floats as changes in the solvent flow rate occur. This is the reason for providing more surge capacity in the column base.

Figure 7 gives results for changes in the feed flow rate. The feed is increased from 1,875 to 2,250 kg/h at time equal 0.2 h. At 3 h feed is reduced back to 1875 kg/h, and at 6 hours it is decreased to 1,500 kg/h. These are all large 20% disturbances. The system rides through these disturbances fairly quite well with two exceptions. First, the purity (xB1) of the water bottoms from C1 drops to almost 96 wt % for the large 20% increase in feed flowrate. This performance can be greatly improved by using a steam-to-feed ratio in this column, as illustrated in the next section.

The second problem is the IPA purity (xD2) in the distillate from column C2 for the 20% increase in feed flow rate. The purity drops to about 99.7 wt %. This occurs because the

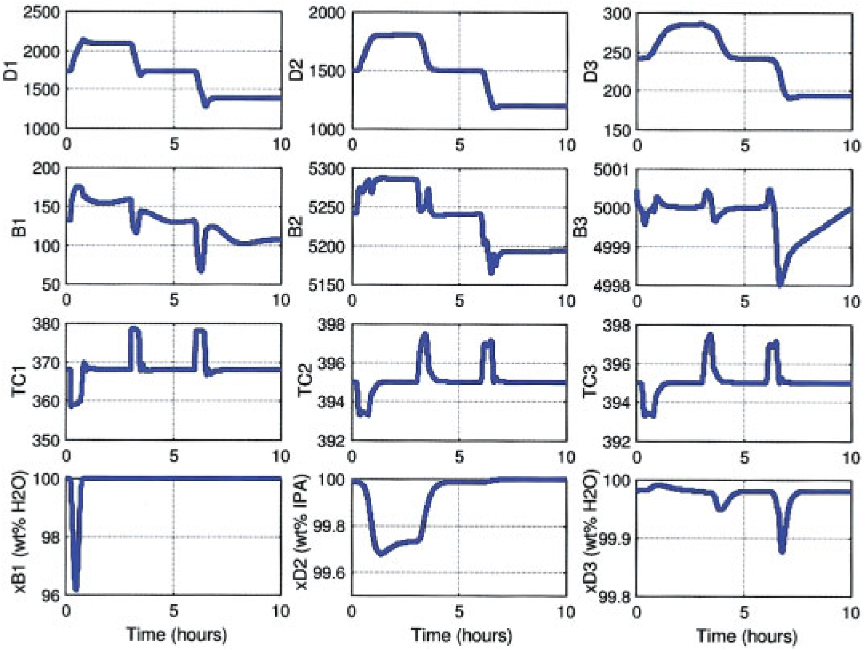

Figure 7. Feed rate changes.

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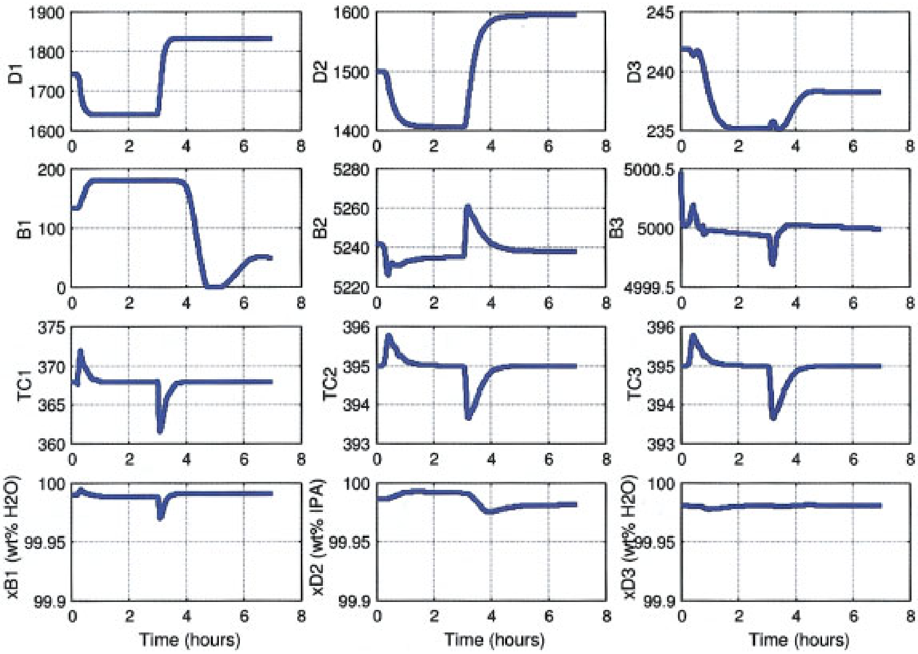

Figure 8. Feed composition changes.

[Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

solvent flow has not been increased. For the decrease in feed flow, the IPA product is overly pure.

This result suggests that the solvent flow should be ratioed to the feed flow. There are several alternative ways to do this, which are discussed in the next section.

Figure 8 gives results for disturbances in feed composition. At time equal 0.2 h the feed is changed from 80 to 75 wt % IPA, and at time equal 3 h it is changed from 75 to 85 wt % IPA. These disturbances are handled well by the control structure.

Modified Control Structure

Ratioing the flowrate of the solvent fed to the extractive distillation column can be handled in several ways. Figure 9 shows the scheme proposed in this article. The basic idea is to always have plenty of solvent. Too much solvent wastes some energy but it improves the separation in C2. Too little solvent can lead to off-specification product.

So if the feed flow is increased, the solvent should be increased *before* the feed flow rate change occurs, that is, the solvent should *lead* feed for an increase in feed flow. However, if the feed flow rate decreases, the solvent should be decreased *after* the feed decreases, that is, the solvent should *lag* the feed for a decrease in feed flow.

These objectives are exactly the same as those in furnace firing controls, where it is necessary to always have an excess of air in the furnace. Here, the air must lead the fuel for an increase in firing rate, but must lag the fuel for a decrease in firing rate.

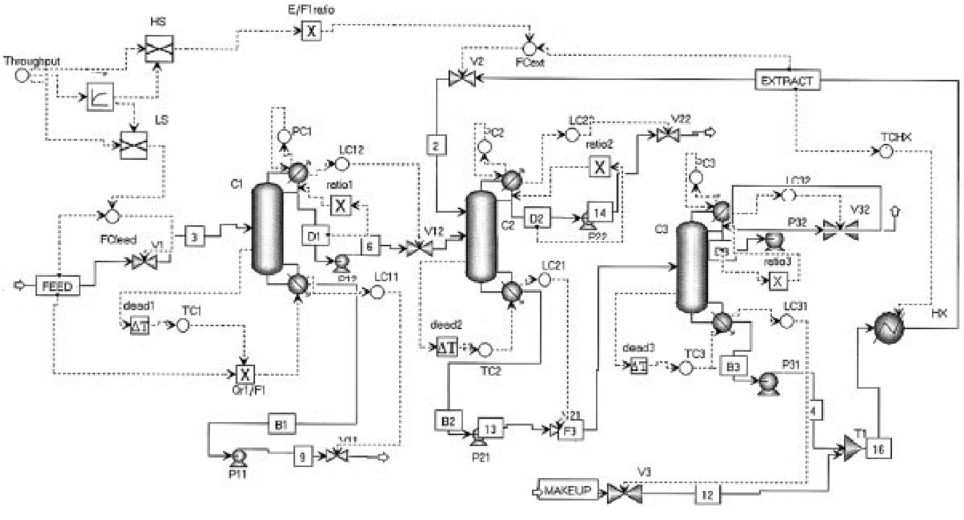

Figure 9. Modified control structure.

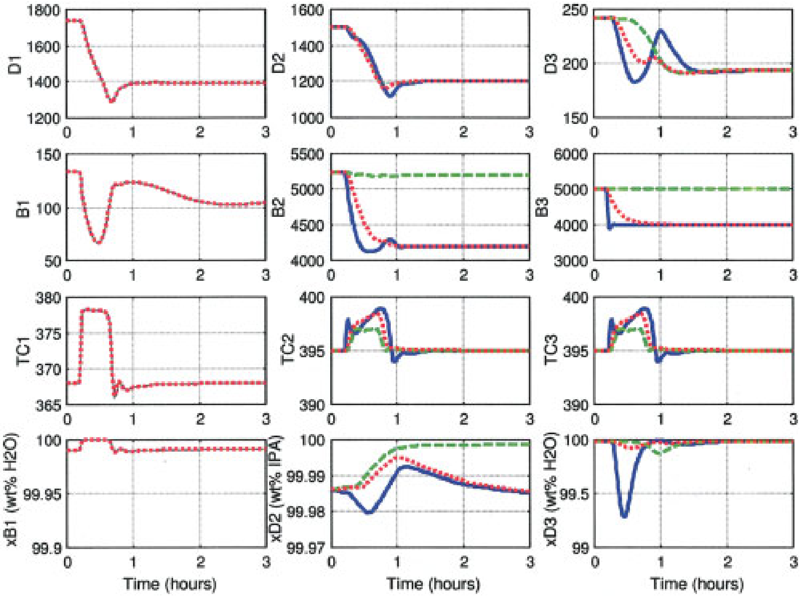

Figure 10. Effect of lag in solvent-to-feed ratio.

[Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

These dynamic objectives can be easily achieved by the use of a first-order lag, a high selector and a low selector. The setup is shown in the upper left corner of Figure 9. It is assumed that an upstream level controller is adjusting the setpoint of the feed flow controller to the three-column dehydration process. The controller output signal from this "throughput" controller is sent to the lag, the high selector (HS), and the low selector (LS). The output signal from the HS is fed to the multiplier ("E/F1 ratio") whose output signal is the set point of the extract solvent flow controller. The output signal from the LS is the setpoint signal of the feed flow controller.

This simple circuitry achieves the desired dynamic performance. For example, suppose the upstream controller calls for an increase in feed flow. The signal from the "Throughput" controller will increase. The HS output signal will immediately increase, which will immediately increase the solvent flow. The output signal from the lag will increase slowly, so the output of the LS will increase slowly. Thus, the feed flow will increase slowly.

To illustrate the different types of responses, consider the results shown in Figure 10. Three cases are shown for a 10% decrease in the feed flow rate. In the first case (the solid lines),

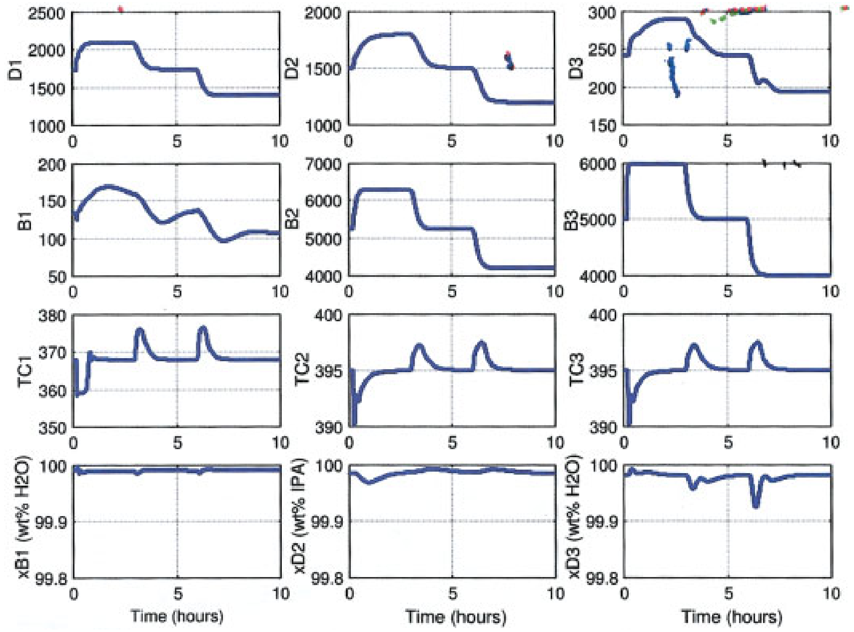

Figure 11. Feed rate changes with modified control structure.

[Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

the solvent flow is decreased instantaneously. This results in a drop in water product purity (XD3) from the top of column C3. In the second case (the dashed lines), the solvent flow is kept constant. The drop in XD3 is greatly reduced, but the IPA product is now over purified (XD2). In the third case (the dotted lines), the solvent flow rate is gradually decreased (using a 10 min first-order lag). These results demonstrate the need for dynamic elements in the ratio scheme.

Figure 11 give results using the modified control scheme for the same sequence of feed flow rate changes. Note that a steam-to-feed ratio has been added in column C1, which eliminates the xB1 purity problem. These results should be compared with those given in Figure 7, for the fixed-solvent flow rate structure. The purities of both the IPA and water products are held closer to their desired values.

Conclusion

The plantwide control of an organic solvent dehydration process has been studied. The specific numerical case is the removal of water from a 2-propanol/water mixture.

A unique control scheme is developed and shown to give effective control in the face of very large disturbances.

Notation

B = bottoms flow rate, kg/h
D = distillate flow rate, kg/h
F = feed flow rate, kg/h
K_C = controller gain, dimensionless
P = pressure, atm
Q_R = reboiler heat input, kW
RR = reflux ratio = R/D
TCn = control tray temperature in column n, K
$xB1$ = purity of C1 bottoms, wt % H_2O
$xD2$ = purity of C2 distillate, wt % IPA
$xD3$ = purity of C3 distillate, wt % H_2O
τ_I = controller integral time, min

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