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## Distill Your Distillation Operations

# TABLE OF CONTENTS

<b>Save Energy In Distillation</b> _____	<b>4</b>
Revamping may cut consumption considerably	
<b>Take A Fresh Look At Your Distillation Columns</b> _____	<b>10</b>
Many towers may benefit from potential improvements	
<b>Effectively Break Azeotropes</b> _____	<b>17</b>
Homogenous and heterogeneous varieties require different methods	
<b>Additional Resources</b> _____	<b>21</b>

# AD INDEX

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# Save Energy In Distillation

Revamping may cut consumption considerably

By John Pendergast, Louisiana State University, Dennis Jewell and David Vickery, Dow Chemical Company, and Jose Bravo, Fractionation Research, Inc.

**S**eparations operations account for roughly 50–70% of the energy used in large-scale chemicals manufacturing [1]. Distillation — which we'll use to encompass the related operations of absorption and stripping — dominates for such separations.

Distillation rules the separation landscape not because of any efficiency advantage but rather because of several other factors that favor it. For instance, distillation:

- scales well, generally to the 0.6 power;
- when performed by itself, doesn't introduce an extra mass agent (solvent, sorbent, etc.) that needs subsequent recovery, as in the case of absorption or liquid extraction;
- allows heat integration within individual units and across facilities, which can

foster effective heat utilization in many separations operations; and

- provides an inter-relationship between pressure and temperature.

Moreover, the technology is well understood and robust, leading to high confidence in designs.

However, its maturity means that distillation usually isn't the focus of academic research, with several notable exceptions such as the work of Rakesh Agrawal at Purdue, Bruce Eldridge, Frank Seibert and Gary Rochelle at the University of Texas, and Ross Taylor at Clarkson. Fortunately, long-established and robust industrial consortia carry out investigations and continue to refine the practice of distillation. For instance, Fractionation Research, Inc. (FRI), [www.fri.org](http://www.fri.org),

which has more than 85 corporate members, operates industrial-scale distillation columns and performs research on modern distillation devices.

Progress is taking place in decreasing distillation energy consumption. Facilities now being planned certainly can benefit. However, energy reductions alone generally can't justify investment to replace an existing column.

Operating companies expect distillation columns to operate for decades; a life span of 30 years isn't uncommon. So, sites over time may make modifications and upgrade instrumentation, and often keep the units in excellent physical condition.

Thus, the most-feasible approach for saving energy is via retrofitting. Here, we'll focus on some ideas for energy reduction in existing columns.

## CONVERTING AN EXISTING SEQUENCE

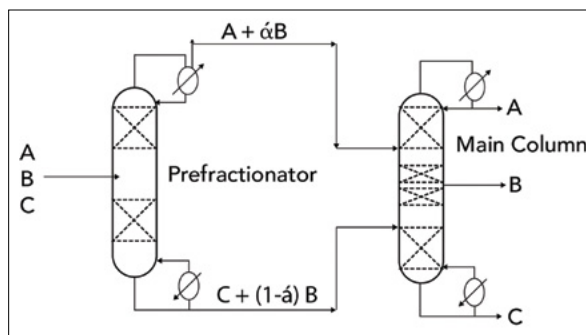
If the infrastructure already exists to perform the separation by conventional methods, you may be able to convert the current sequence to a prefractionator or Petlyuk arrangement (Figure 1). This can save a significant amount of energy for the required separation; typically, it reduces energy consumption by 20–40%. Alternatively, you can produce more products from the facility for the same amount of energy.

Converting an existing facility from a conventional sequence to a prefractionation one certainly isn't trivial; it requires careful study, including rigorous simulation, and then a detailed evaluation of the mechanical modifications necessary. You should assess, e.g., the internal tower hardware before and after the modifications, suitability of the pre-fractionator reboiler and condenser in the new service, turndown capability in the new service, and other details.

A further way to achieve or increase energy savings is by converting the sequence to sequences that employ side-rectifiers or side-strippers.

## OPTING FOR A SINGLE DIVIDING WALL

A dividing wall column (DWC) may save a substantial amount of energy. (Check out, "Consider Dividing Wall Columns," <https://goo.gl/sUY27t> [2].) Depending on whether significant changes in product mix or capacity



### PREFRACTIONATOR

Figure 1. Adding such a column typically leads to a 20–40% cut in energy consumption.

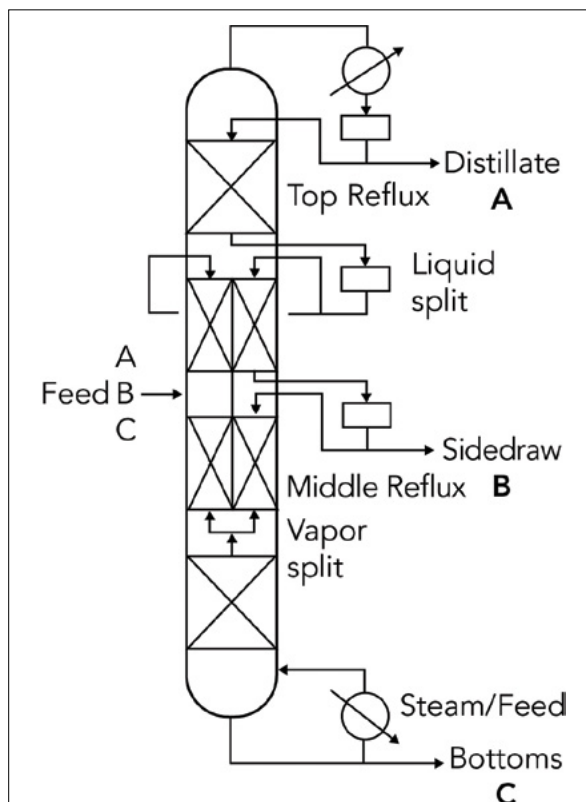
## You must analyze the system individually, taking into account the utilities and economics for the particular operation or entity.

requirements have occurred over time, it may be possible to convert a two-tower system to a single dividing wall column (Figure 2). This is more likely when the original tower configuration was developed to remove relatively small fractions of light component (A) or heavy component (C) or both.

You must assess mechanical details such as tower hydraulics, feed locations and auxiliary equipment rating as well as other details to determine the feasibility of such a conversion. As with all complex column configurations, it's essential to remember that the levels of energy consumption are an important consideration for the evaluation of the benefits of new separation configurations. While the single DWC will consume less energy from a First Law perspective, it may be disadvantaged from a Second Law perspective. So, you must analyze the system individually, taking into account the utilities and economics for the particular operation or entity.

### POWERFUL LOW-COST “TRICKS”

Small changes that require minimal capital investment sometimes can produce great benefits. You should consider such “tricks” when searching for improved energy or



#### DIVIDING WALL COLUMN

Figure 2. Existing towers with low-volume overheads or bottoms often are good candidates for conversion.

capacity performance from an existing facility. Some examples include:

- *Feed location.* Always look into this. A feed in less-than-optimum position in the distillation column negatively affects energy use and, thus, capacity. Feed location might have been appropriate initially but, as time has passed and feed

or product specifications have changed, may require repositioning. A small change in feed location can save on large reboiler duties.

- *Use of side-draws.* A precursor of the concepts of prefractionation and the DWC is the thoughtful use of side-draws in a given multicomponent distillation train. A classic example is that where top and bottom products are needed at high purity and the middle boiler is present in small amounts in the feed. If you look into the composition profiles of existing columns such as these, you'll see that the concentration of the middle boiler presents a steady-state "bulge" that reduces at the ends. Using a side-draw within that bulge allows retrieving the middle boiler at high concentration in a small stream, drastically reducing the heat duty of the column.
- *Treating pressure as a variable.* Engineers generally think of the operating pressure of a distillation column (i.e., the average between top and bottom) as a given. However, you can effectively use changes in pressure to alter performance. Because relative volatilities vary with pressure, it sometimes proves useful to lower column average operating pressure to require less heat duty. A very common example of this is the well-known trend in refineries to use low-pressure-drop packing in crude and vacuum towers instead of trays to reduce pressure drop and improve fractionation.

- *Feed condition.* Adding or removing heat from the feed can change the requirements of a reboiler or a condenser. This may not alter the overall heat balance but might allow for easier heat recovery schemes with feed exchangers that may help with the loads on reboilers and condensers.

Properly assessing the viability of these modifications requires a well-validated model of the distillation column that can provide reliable pressure, composition and temperature profiles.

## HYBRID OPERATIONS

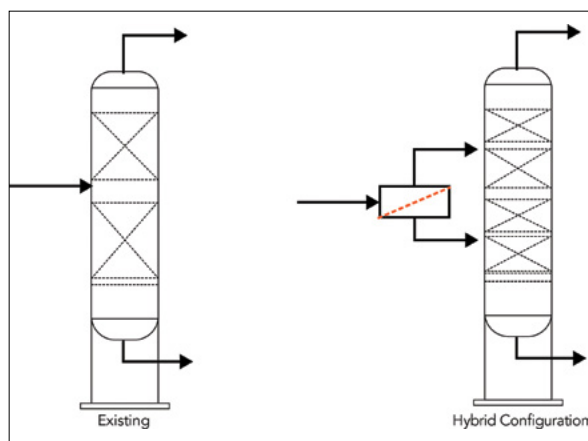
Progress in both membrane and adsorption materials has enhanced the prospects of hybrid separation, by which we mean the use of either membranes or an adsorber in tandem with distillation. The combination of these unit operations potentially can reduce the separation energy of some energy-intensive separations while still meeting the financial hurdles required of any improvement project.

While myriad combinations of other operations with distillation are possible, these two have a strong synergy with distillation that can lead to improved energy efficiency or capacity.

*Distillation plus membranes.* Membranes are well suited for pre-fractionation; so, they may offer an attractive option for low energy separation upstream of the distillation tower.

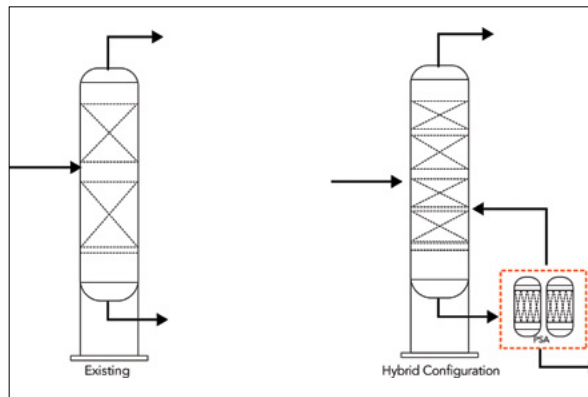
Figure 3 provides a simplified representation of the concept. (The graphic shows a tower with packing but the concept applies equally well to trays.) The permeate and retentate from the membranes then can go to the optimum location in the tower, lowering the energy consumed by a substantial fraction. As pointed out by Sholl and Lively [3], the use of membranes to pre-fractionate light olefin from paraffin might reduce the energy requirements by a factor of 2 to 3. As always, the key challenge to any large-scale separation with membranes is the surface area required. Membrane separations scale directly, while, as noted earlier, distillation scales to the 0.6 power, which drives large-scale separations to distillation. Applying membranes in tandem with distillation doesn't change the scale factor but does eliminate the need for the membrane to provide pure product; this enables selecting the membrane area that gives the optimum level of performance within economic constraints.

*Distillation plus adsorption.* Such a system holds promise for lowering the energy consumption for separating zeotropic mixtures. Indeed, this hybrid operation already is widely used for breaking the azeotrope between ethanol and water — it may well be the preferred method of operating assets producing anhydrous ethanol. As shown in the simplified schematic in Figure 4, the light components are allowed to “slip” to the bottom of the tower, from which they go to the adsorber where light components



#### MEMBRANE/DISTILLATION HYBRID

Figure 3. Using a membrane as a pre-fractionator frequently can provide substantial energy savings.



#### DISTILLATION/ADSORPTION HYBRID

Figure 4. An adsorber can enable reintroducing a light component at an optimum location in a column, lowering energy consumption.

are captured and returned to the tower at the optimum location.

While the figure illustrates pressure swing adsorption (PSA), there's no fundamental reason why a hybrid process couldn't use temperature swing adsorption (TSA). The ability to rapidly regenerate beds with pressure swings, thus reducing the amount and size of the adsorbent beds needed, generally drives selection of PSA over TSA.



## RETHINK YOUR CONFIGURATION

As we've pointed out, retrofitting existing columns with sequences such as Petlyuk processes, DWC, side strippers and side rectifiers and other complex arrangements may conserve distillation energy. In addition, progress in both membranes and adsorption materials has opened up opportunities for applying hybrid unit operations that may well fit into existing infrastructure and produce acceptable economic returns on energy reduction opportunities.

Modern simulation tools and optimization methods enable investigating all the potential alternatives for distillation separation sequences and selecting the best sequences. They play a key role in the design of new facilities but also can help in revamping existing columns where energy savings alone won't justify a replacement unit. Perform such evaluations for those revamping options that seem appropriate for your plant; you may identify opportunities for substantial reductions in energy consumption. ●

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# Take A Fresh Look At Your Distillation Columns

Many towers may benefit from potential improvements

By Seán Ottewell, Editor at Large

**O**pportunities abound for enhancing the performance of tens of thousands of distillation columns in use worldwide today. However, that requires looking at every aspect of their operation, maintenance and management, say experts.

Jose Bravo, President of Fractionation Research Inc., Stillwater, Okla., points to a variety of energy saving measures that have been — and still are being — implemented in distillation systems worldwide. They encompass three broad areas: process integration, peripherals, and advanced control and optimization.

Process integration can take advantage of a range of technologies. For instance, he

cites dividing wall columns and notes that hundreds of such columns now operate at chemical plants and refineries. (The article, “Consider Dividing Wall Columns,” <http://bit.ly/2OgwW6a>, has more details on the technology). He also mentions several dozen projects for recovering heat from condensers to preheat feed, a similar number involving lower pressures in main fractionators at refineries, and the use of advanced packings and re-traying to reduce pressure drop.

The second category includes developments such as enhanced surface tubes for condensers and reboilers. In the third group, he counts technologies such as smart controls, adaptive controls, model-based controls and on-line continuous optimization.

Importantly, however, he notes that energy alone usually isn't a large enough economic incentive.

"All these projects are always associated and justified with other drivers as well, energy being only one. The others are, generally, lower capital for expansion or new capacity, safety or reduction of carbon dioxide footprint. Combined, they often give a positive economic outlook that allows the plants to invest," he emphasizes.

## **IMPROVING EXISTING OPERATIONS**

Henry Kister, senior fellow and director of fractionation technology for Fluor, Aliso Viejo, Calif., has experience with all these drivers. However, one crucial issue often overlooked is making the most out of existing equipment, he notes.

"Troubleshooting, revamping and eliminating waste can offer huge benefits in capital investment, downtime, carbon dioxide emissions and energy use. Unfortunately, the attention paid to this resource in the energy-saving literature has been too little to reflect its tremendous potential," he stresses.

Kister points to a broad range of examples in Norman Lieberman's book "Process Engineering for a Small Planet: How to Reuse, Re-Purpose, and Retrofit Existing Process Equipment" as a perfect demonstration of how subpar engineering, poor

troubleshooting, and wasteful practices guzzle energy, generate carbon dioxide, and waste the earth's resources.

In one, Lieberman describes a case in which modifying trays and downcomers in a fractionator as well as adding mist injection to the overhead compressor could have circumvented erecting a giant new fractionator with a new oversized overhead compressor. "Just the compressor oversizing was estimated to waste the amount of crude oil that 400 families use daily. Fabricating the new steelwork and structures consumed immense amounts of energy and emitted tons of carbon dioxide, all of which were unnecessary," says Kister.

In another, Lieberman recounts how he was tasked with designing a new, \$4-million pre-flash tower to replace an existing one that experienced flooding. "Instead, he spent one day eliminating the flooding in the existing tower by reducing the bottoms liquid level (high levels caused flooding), and blowing the level taps on the reflux drum water draw-off boot, unplugging them to prevent refluxing water which caused emulsion and flooding on the trays," Kister points out.

A third example he mentions involves Lieberman being tasked with designing another new tower to recover diesel from the bottoms stream of a refinery crude fractionator. Instead, he opted to troubleshoot, measuring zero pressure drop across the fractionator bottom trays, and

## Such long-term issues waste huge amounts of time, money and manpower.

observing that stripping steam rates did not affect diesel recovery. “Both indicated missing stripping trays. Repairing the trays fully solved the problem and modifying the steam entry prevented recurrence, circumventing the unnecessary new tower with its steelwork, energy waste and associated carbon dioxide emissions,” Kister adds.

“Lieberman presents a book full of similar experiences, with different degrees of management support to his troubleshooting endeavors. In the examples cited above, managements set their minds on the new column solutions in preference to troubleshooting: jack hammers to crack nuts,” notes Kister.

Kister’s own book “Distillation Troubleshooting” illustrates similar issues. For example, one case study describes an olefins debutanizer receiving two feeds. The larger feed contained only 8% C4s and was the bottoms of a stripper that removed C3s; with good design that column also would have stripped the 8% C4s. When at one time the larger stream was bypassed around the debutanizer, the steam consumption dived to less than half. As Kister notes: “Whatever does not enter the column does not consume energy.”

Another describes how direct-contact compressor intercoolers provided poor cooling for many years, causing excessive compressor energy consumption. This only was noticed after plant rates were raised and the inadequate cooling began restricting throughput. A close review showed that the tower’s pipe distributors had a quality rating index as low as 13% (>75% is good). Debottlenecking with well-designed, inexpensive spray distributors and improved shed decks reduced compression energy and debottlenecked compressor capacity.

Such long-term issues waste huge amounts of time, money and manpower — as further illustrated by the case of a refinery where for 11 years about 2% of the crude oil that should have been recovered as valuable diesel product ended up in the much-less-valuable residue from a crude tower. This also wasted energy because the diesel in the residue was vaporized in a downstream fired heater. A simple water leak test at the turnaround revealed that the diesel draw pan was leaking; seal-welding the joints recovered the lost diesel yield. “Excellent troubleshooting,” comments Kister.

Then, too, there's the case of a retrain project at closer spacing that increased tray count by 50%. This successfully improved product purity — but the new trays operated right at their capacity limit. Because the plant wanted to raise throughput by 15% at the next turn-around, engineers planned to replace the new trays with structured packing. The tower was large, so the replacement would require tons of steel and lots of money. Getting the energy balance to close by solving flowmeter issues revealed that the reflux and reboil were much higher than design, meaning the trays fell well short of achieving their design efficiency. “Modifying the trays solved the problem without the extra steel and expense of the structured packings,” Kister explains.

“In last three cases, managements supported making the troubleshooting solutions: there was no need for sledge hammers,” he emphasizes.

In another experience, a small packed stripper tower was erected to purify a wastewater stream destined for cooling water makeup. The entering wastewater poured above the hats of the liquid distributor. The oversized hats filled most of the cross-section area, leaving little area for vapor ascent simultaneous with liquid descent. The resulting high vapor velocities between the hats blew the incoming water upwards, causing flooding. “Two different consultants studied the problem, the first offering an incorrect diagnosis and

an ineffective fix, the second, based on an extensive simulation study, incorrectly concluding that a bigger tower was needed. The plant gave up and junked the tower, with wastewater still going to sewer. Good troubleshooting, and a nickel-and-dime job of modifying the hats and feed pipe, would have produced good water,” he points out.

“Finally,” says Kister, “a new hydrocarbon gas absorber (not by Fluor) flooded at reflux rates exceeding two-thirds of the design due to premature downcomer choke induced by excess foaminess. Based on this diagnosis, we were requested to provide new trays with enlarged downcomers. Our response was: ‘Do we have to?’ Performance is judged by product purity, not reflux. Upon adequate testing, the overhead gas impurity was found tenfold below design. The absorption was good thanks to the colder-than-design reflux. Here the ‘don't worry, be happy’ approach, based on good troubleshooting, saved resources, energy, and money.”

## PROCESS SIMULATION SUCCESSES

The experiences of KBC Advanced Technologies, London, a Yokogawa company, amply illustrate the potential benefits of Bravo's third category, process control and optimization.

When considering energy in distillation revamps, process simulation built on rigorous thermodynamics, such as KBC's Petro-SIM,

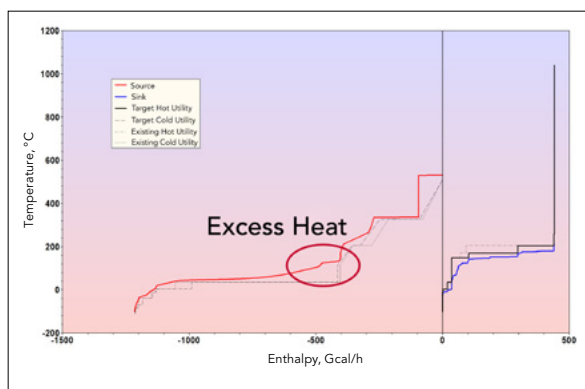
should serve as the fundamental tool for evaluating revamp options, stresses KBC business development executive Andrew McIntee.

“This type of tool can be used to assess all the engineer’s options and calculate the potential for making improvements. Adding heat pumps, side condensers/reboilers and low-grade heat recovery can be fairly straightforward; however the challenge is that there are many options, and the best solution is usually very specific to the application,” he explains.

Pinch analysis enables strategically evaluating options, he notes. These could involve, for example, modifying column pressures to fit in with the background heat availability, and investigating if a heat pump brings any net benefit if it doesn’t recover heat across the pinch. In addition, column pinch analysis allows systematically looking at feed location, temperature and side reboiling/condensing.

“Using powerful and accurate tools, users can consider all the interactions of the column and its equipment with the surrounding asset and complex. Combining these unlocks greater potential than local optimization,” adds McIntee.

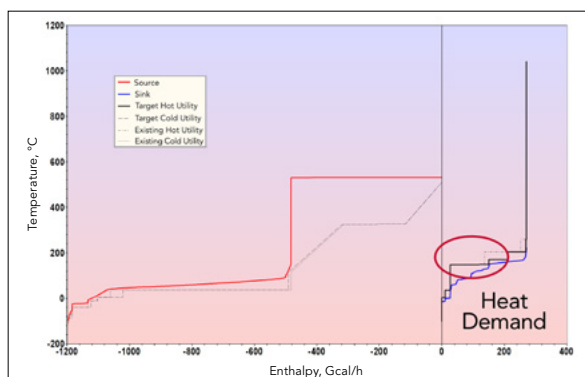
As an example, he cites the experience of a client wishing to optimize integration to save energy and capital at a world-scale



#### SURPLUS LOW-GRADE HEAT

Figure 1. Complex 1 had the potential to supply very low pressure steam and hot water.

Source: KBC.



#### HEAT DEFICIT

Figure 2. Complex 2 had a large demand for low grade heat. Source: KBC.

integrated petrochemical site. Two large facilities there (Complex 1 and Complex 2) had been optimized individually — but TotalSite pinch analysis revealed many valuable opportunities for integration.

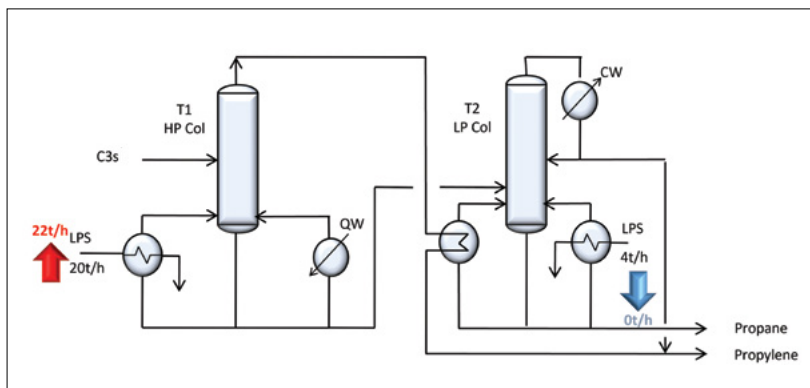
For example, the analysis found that Complex 1 potentially could supply very low pressure steam (VLPS) and hot water (Figure 1). However, that facility didn’t have much demand for these. Meanwhile, in Complex 2, Cracker 2 lacked sufficient supplies of low-grade heat (Figure 2).

Complex 2 had been designed with a heat pump on the C3 splitter. Seen in isolation, this was a rational design because Cracker 2 had a heat deficit and could use a heat pump to recover energy from the condenser.

However, the integrated design utilizing hot water from Complex 1 provided

both operational savings (6 MW for the heat pump compressor) and capital ones (eliminating the cost of the compressor).

“Overall, the complex was able to generate 22MW of power from low-grade heat, while



### PROPYLENE SPLITTER

Figure 3. Analysis led to a recommendation for a shift in duty between the two towers. Source: KBC.

revised driver selection improved steam balance and reduced capital costs.

An operational quick win was identified through examining the whole distillation train. Modelling it allowed clear assessments of the potential energy saving operation while ensuring the process was not compromised,” says McIntee.

Another example involves a propylene splitter (Figure 3) where a shift in duty between towers was recommended.

Here, C3 separation columns were well integrated with overheads from the first, high-pressure tower (T1), used to reboil the second, low pressure column (T2). Heat input to T1 was used twice. The analysis showed that an increase in duty (supplied by low pressure steam, LPS) in the T1 reboiler reduces the duty of the T2 reboiler by factor of two. This led to a recommendation to maximize LPS use on T1. The overall saving is 2 t/h of LPS worth

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## Digitalization promises new technologies in the area of distillation energy efficiency improvements.

\$450,000/y; a plant trial has confirmed this saving.

A third called for resequencing of debutanizer columns. Here, the original design called for steam reboilers on De-iC4 + De-C4 columns. Due to significant change to alkylation feedstocks and rate during the front-end engineering design, the energy use and, therefore, operating cost rose by nearly 160%.

Pinch analysis and considering the entire plant including the utilities showed a resequencing of the columns (from De-iC4 + De-C4 to De-C5 + C4 splitter) could reduce overall energy use.

The first column utilized excess high-pressure steam while the second column could be fitted with a heat pump.

“Overall energy use returned to near original levels with the cost savings totalling \$27.6 million/y,” says McIntee.

Digitalization promises new technologies in the area of distillation energy efficiency improvements, he believes. For example, digital twins and improved data analytics directly integrated with closed-loop advanced process control can identify unusual column operation.

“KBC is completing proof of concepts with multi-period proactive optimization, where energy cost is optimized over a time period. For example, the process is adjusted so that low-priced energy can be stored for better financial benefit later on,” he notes.

“On the grassroots design front, Pinch fully integrated into simulation is almost here, and the next steps are for AI [artificial intelligence]-driven option evaluation and opportunity identification to allow continual optimization by considering all the other changes happening during the design. This moves the process from a linear step to continual iteration and a global optimum,” he concludes. ●



# Effectively Break Azeotropes

Homogenous and heterogeneous varieties require different methods

By Tom Schafer and Alan Erickson, P.E., Koch Modular Process Systems, LLC.

**A**n azeotrope is a complex formed by two or more chemicals and has the same composition in the liquid and the vapor phase. This interesting property means that the compound mixture distills without separating. Some common well-known binary azeotropes are ethanol (95.4%) and water (4.6%) as well as diethyl ether (33%) and halothane (66%), formerly used in anesthesia. However, as sometimes happens in chemical processing, an azeotrope may be formed unexpectedly or, even if recognized, may make downstream processes additionally challenging.

This article will discuss some methods used to “break” azeotropes that may exist in binary or ternary forms and their applications, with a focus on breaking azeotropes using various distillation methods. Solvent

recovery often necessitates breaking azeotropes when substances are required in a purer form, either downstream or in other areas of the chemical process. Water removal may motivate others. Situational and contextual factors also may impinge on process optimization. Several basic types of processes to break azeotropes using distillation and real-world examples are described.

## **DISTILLATION AND DECANTATION**

The easiest azeotrope to break is a heterogeneous azeotrope. Unlike homogeneous azeotropes, heterogeneous azeotropes' two compounds are not totally miscible. The combination of distillation and decantation works with heterogeneous azeotropes whereby condensation of the azeotrope results in two liquid phases that then can be

decanted. That is, each of the two phases can be fed to separate stripping columns. Examples of mixtures typically separated this way include the minimum boiling azeotrope of n-butanol and water or dichloromethane (methylene chloride) and water.

In the example of n-butanol and water, after condensation, the water-rich phase goes to a stripping column where butanol-free water comes out the bottom and the butanol-rich phase goes to another column where dry butanol comes out the bottom and the distillate of each column approaches the azeotropic composition. Generally, chlorinated hydrocarbons and water or any of the aromatic or paraffinic solvents and water can be separated that way.

## PRESSURE SWING DISTILLATION

When two substances are totally miscible, as in homogeneous azeotropes, other factors can come into play. Pressure swing distillation is effective when the azeotrope's composition is a reasonably strong function of pressure. An azeotrope distilled at a low pressure subsequently is fed to a second distillation column operating at a higher pressure where the azeotropic composition is substantially different. Acetonitrile and water and tetrahydrofuran (THF) and water are two typical azeotropes broken using this method.

At atmospheric pressure, the azeotrope formed between THF and water is 95% THF. However, at 95 psig, the mixture is closer

to 88% THF. By operating two columns at the different pressures, dry THF can be retrieved from the bottom of the 95 psig column and the azeotrope emerging at the top, recycled to the atmospheric column.

## AZEOTROPIC DISTILLATION

This distillation process is used to break homogeneous azeotropes by introducing an additional component called an entrainer, which forms a lower boiling decantable ternary azeotrope. The ethanol and water azeotrope (95.6:4.4) mentioned above is a classic example of this type of process whereby benzene (or the more commonly seen cyclohexane) is used as the entrainer. Similarly, an isopropanol and water azeotrope can be broken using di-isopropyl ether as the entrainer. The lower boiling azeotrope then is condensed and decanted.

Typically, this process requires three separate distillation columns. The first column distills the alcohol to the azeotrope with water removed as the bottom product. The second column is the azeotropic column, in which the alcohol is removed at the bottom and the ternary azeotrope is removed as the distillate product. This distillate then is decanted with entrainer-rich phase and returned to the azeotropic column and the water-rich phase is sent to the third column to remove entrainer and alcohol from the water. Sometimes, columns one and three can be combined.

## In the case of ethanol and water, ethylene glycol may be used to change the behavior of the two substances and allow distillation to be effective.

Obviously, the use of specific entrainer substances will not be applicable to all industries. Introduction of a known toxin or carcinogen such as benzene, for example, is a nonstarter for a pharmaceutical process.

### **EXTRACTIVE DISTILLATION**

In contrast to azeotropic distillation, in extractive distillation an additional high boiling component is introduced. The added component, which basically is a diluent, changes the original binary pair's behavior such that the azeotrope can be broken. Azeotropes of ethanol and water or methanol and acetone can be broken this way. Azeotropes of refrigerants also are amenable to breaking using this method.

In the case of ethanol and water, ethylene glycol may be used to change the behavior of the two substances and allow distillation to be effective.

### **ALTERNATE TECHNIQUES FOR BREAKING AZEOTROPES**

Liquid-liquid extraction (LLE) can be used if the difference in the solubility of a solute

between two liquid phases breaks azeotropes. Pyridine-water and THF-methanol are examples of azeotropes that can be broken via LLE.

Newer membrane technologies — in which the membrane material is robust enough to withstand attack by the substances being separated and the pore size can distinguish the complexes formed by a mixture's different components — also can break azeotropes. However, membranes also are subject to clogging. Over time, even trace amounts of solvents or particles, such as minute amounts of catalyst, can compromise the polymeric material used in membrane filtration.

### **DETERMINING THE BEST APPROACH**

Breaking azeotropes or even recognizing when they are present is not always straightforward. Most undergraduate chemical engineering curricula do not spend much time on nonideal separations in their mass transfer classes. Turning to companies with expertise in mass transfer and separations is critical, particularly when there is a suspicion

## It takes a substantial amount of experience to develop and conceptualize the proper approach.

that azeotropes may be in play. It takes a substantial amount of experience to develop and conceptualize the proper approach.

Pilot plant testing often is required to test the actual feedstock and show that the conceptual design first offered will meet required specifications. The drums of actual feedstock that customers send to companies for pilot scale testing frequently contain minute amounts of other contaminants, which differ from the ideal feedstock upon which the concept design was developed. This can affect the path chosen to

break the azeotrope, and pilot testing can uncover those trace components and find solutions.

Working with or around nonideal separations, such as azeotropes, may require multiple distillation columns and several decades of chemical process design experience.

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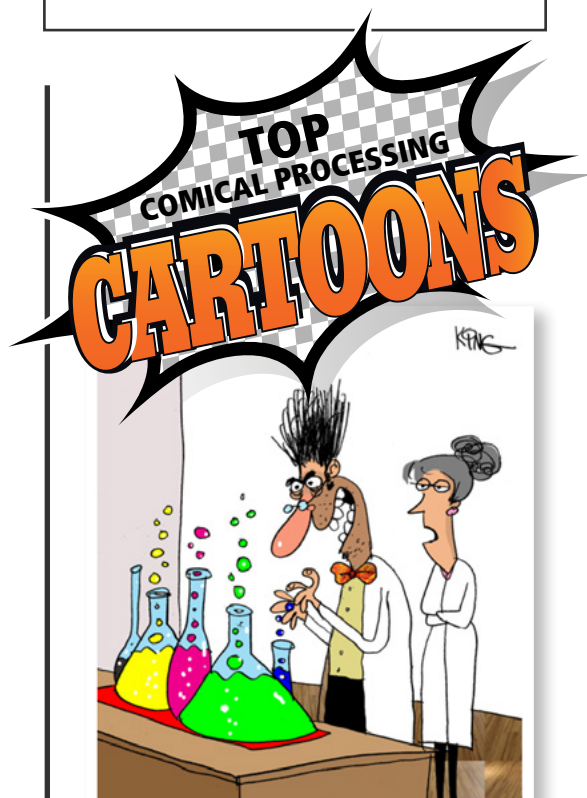
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