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OPTIMIZE DISTILLATION COLUMN REVAMPS

Optimize Distillation System Revamps

Revamping can improve most existing columns and, indeed, provides one of the most economically attractive opportunities available in process engineering today. Use this proven strategy to find the most-cost-effective option.

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Distillation remains the predominant separation technique in the chemical process industries (CPI), accounting for more than 90% of product recovery and purification applications. Its use spans the entire CPI, from massive continuous chemical and petroleum complexes to small batch facilities manufacturing specialty chemicals such as materials additives, pigments, paints, inks, pharmaceuticals, flavors, and fragrances. With more than 40,000 distillation columns in operation in the U.S., the capital investment involved exceeds $8 billion *(1)*.

Though revamping of distillation systems is not a new concept, it is assuming greater importance today. Many companies now are trying to maximize their return from existing sites, rather than investing in new grassroots facilities. Plus, the opportunities for column revamping abound: An increasing number of distillation systems are required to process variable-feed-composition streams and to meet more-stringent product and byproduct specifications. Many batch systems are operated in campaigns, which may make additional demands as the feed components and composition are perpetually changing. Improving the performance of such processes promises a high economic return. In fact, for most distillation systems currently in operation, a revamp may provide significant improvements in the competitive position of the process.

Revamping opportunities are present in most existing distillation systems, with potential improvements including:

- eliminating operational bottlenecks and increasing capacity;
- implementing a new process or product within existing equipment;
- enhancing product purity, thereby creating possible new markets for an existing product;
- boosting product or raw-material recovery;
- purifying raw materials;
- reducing waste-disposal quantities; and
- removing and capturing materials from water and air waste streams.

Even though these factors make revamping a worthwhile endeavor, the distillation process sometimes is not considered a part of the primary production line but instead a low-priority support operation. Consequently, too often, it does not receive the emphasis and attention it deserves.

Distillation for recovery of solvent or unreacted raw materials also typically is considered a lesser operation than product recovery. For such "secondary" operations and within smaller facilities, financial constraints can be particularly sensitive, especially if it is assumed (mistakenly) that revamping the distillation column will be prohibitively expensive.

Consider a real revamp

Many companies don't actually assess the revamping of an existing unit — not realizing that a high economic return usually is easily achievable with a well designed and executed revamp. Instead, the

traditional "revamping" approach often taken is to design and build a new column, rather than to work within the confines of the existing unit. This may involve lower risk, but it almost always is more costly in terms of capital investment (including for equipment that a well-designed revamp may obviate) and lost production due to downtime.

The reasons why improvement of existing equipment doesn't get the emphasis it deserves are many and varied — but they generally boil down to a lack of solid understanding of what can be accomplished and a lack of realistic analysis of capital requirements. Yet, to reiterate, revamping usually is the most cost-effective solution.

Revamps afford the opportunity to take advantage of the significant progress recently made in distillation systems. Such developments include the commercialization of higher-efficiency trays and packings that promise high economic returns. Today's high-performance mass-transfer internals offer dramatically reduced column pressure drops and increased turndown ratios — enabling operation over a wider range of high and low liquid-flow loadings — than was previously possible.

The purpose of this article is to show production and process engineers how to determine, given their particular objectives, whether or not revamping is a viable option. It introduces an approach that can be applied regardless of scale of operation or type of product. We will not get into specific design practices, which can be found in several excellent theoretical and practical texts such as Refs. 2–8. Instead, we will outline the following:

1. How to achieve a successful revamp;

2. How to factor in environmental issues;

3. How to develop the right strategy — including assessing the desired operating conditions, finalizing design parameters, and selecting the proper equipment; and

4. How to analyze the economic impact;

In addition, we will provide case histories of distillation system revamps to illustrate the value of the approach.

In short, this article will provide an overview of all the factors that must be considered when evaluating a possible distillation-system revamp. Though the recommended approach involves more upfront effort by the production engineer, it will identify the most economical way to achieve operational objectives.

HOW TO ACHIEVE A SUCCESSFUL REVAMP

One of the main issues involved in revamping a distillation system is determining what must be done to effect an economic gain for the plant. We'll outline several steps that can serve as a guide.

Define system objectives

The first step is to define the desired objectives of a revamp. Information must be gathered relating to the processing requirements, including determining the full range of potential feedstock compositions and stream conditions. It also is helpful to note how many product lines are campaigned upstream of the distillation system. The desired minimum and maximum processing capacity also must be defined, as well as the product composition wanted (both the minimum acceptable and "ideal"). It also may be necessary to specify other product attributes such as color and clarity. A report of the product specifications and distillation system requirements should be confirmed with the organization's Marketing Product Manager and the Production Manager, respectively.

Evaluate existing operations

The next step is to assess how the existing distillation system works. On-site observations are crucial to the understanding of the current system's operation — they often can identify whether existing equipment, instrumentation, and controls actually may have the potential to achieve the necessary results with only minimal modifications. All process and instrumentation diagrams (P&IDs) and controls should be personally field-verified. In short, field assessment is not only an excellent method for understanding current operations and problems, but also is a cost-control measure that may save a company from pursuing unnecessary expenditures.

Visual inspection — If the time frame of the project allows, visually inspect the column internals by observing the operation through the accessways or handholes. Look for internals that may have been displaced or those that have become misaligned over time. Also, check for plugging within the column, as well as deformation or any signs of excessive wear of packing material or distributors. Surfaces that have become fouled or show stress cracking or discoloration may indicate such problems as poor wetting, pressure surges during operation, or excessive flashing near the reboiler discharge. Additionally, if column performance has declined over time and signs of materials corrosion are apparent, consult a metallurgist. A competent metallurgist can assess the impacts of the current corrosion and specify either operational conditions less corrosive to the existing materials or materials that offer a higher degree of corrosion resistance.

Radioactive tools — Three established column on-line diagnostic tools involving the use of radioisotopes — grid scanning, radioactive tracing, and computer-assisted tomography (CAT scans) — can be useful in assessing the hydrodynamics of the column under existing operational conditions. The internals deficiencies identified then can be addressed in the revamping design.

Grid scanning basically is a measurement of the column cross-sectional density profile via a series of

systematic gamma scans taken through the column vessel. The technique exhibits limitations on columns diameters less than 18 in. or more than 30 ft, and on very dense systems. Grid scanning can be effective for column integrity checks and the identification of column maldistributions, flooding, foaming, and entrainment.

Radioactive tracing introduces a traceable material into the process and tracks its passage within the system with detectors. Commonly used radioisotopes are bromine 82 and iodine 131 for organic and aqueous systems, respectively. Tracing can be effective for flow-related problems such as holdup, mixing, and leakage.

CAT scans develop detailed cross-sectional density profiles via several angular measurements at fixed elevations. CAT scans are more detailed and costly than grid scans and, therefore, often are used as a detailed complement to a grid scan in identifying specific patterns of maldistribution.

Bowman provides a comparison of radioisotope techniques and their use for maintenance, troubleshooting, and optimization *(9,10)*.

Historical performance — Compare the current system performance with data generated at the time of installation and startup. Can the existing column operate at the same pressure profile, or are there obvious indications of changes to the column internals such as corrosion, misalignment, or blockages? Note any changes and what caused them.

To evaluate current column performance, you will need to review the operations log and any archived charts and data sheets. Look at the maintenance records of the column and ancillary equipment. Verify the mass balance, including the feed, distillate, and bottoms rates and compositions, and reflux ratio, as well as the energy balance, including the condenser and reboiler conditions and utility usages. Is there consistency with the mass balance? Examine the column temperature profiles and their stability to determine if there are any abnormalities in the pressure drop that would indicate column leaks, steam losses, or feed surges. Is the system constrained by the supply rates or condition of the utilities? Does a batch system plateau or get stuck at some point? Check also for any thermodynamic or kinetic effects, such as the onset of a reaction, or a phase change that may cause the conversion of material into waste.

Interview the operators to determine other critical information. Ask them how often the unit must be shut down, and whether this is due to a lack of distillation feed or a product changeover. Does there seem to be a lack of operating staff? Do distractions by other plant activities interrupt the distillation system operation? Do the operators notice problems with fouling or plugging of any equipment or lines in this area of the plant? If so, how do they remedy them?

You also will need to determine *actual* startup and shutdown procedures for the unit. These may identify limitations of the system and additional sources of waste generation. Note any unusual events in the distillation system's history and what may have caused them. Ask the operators to detail when the product streams are off-specification and when the system is performing at its best. They may have information that is key to determining the conditions that lead to column instability.

What is the current instrumentation, and how closely do the system parameters follow the control system? In your inspection of the existing instrumentation and controls, determine if any of the instruments need recalibration. It is not uncommon for distillation systems to perform poorly due to faulty thermocouples or dry thermowells. Also, find out how much of the operation is truly automatic vs. manual, and how often the operators must interrupt the control scheme to use manual settings. If the system is not steady state, how does it change and how smoothly does that happen?

Throughout your observations, address the possibility of the current hardware accomplishing the desired objectives, either by using a different operating algorithm (controls), by correcting an existing malfunction within the system through modified instrumentation, or by modifying or replacing an equipment component. Controls and ancillary equipment revamps/changes often present themselves and enable the company to avoid expensive revamping designs.

For instance, it may be beneficial to modify the control algorithm to increase the liquid or vapor rates by more aggressive condensation and boilup rates, and to incorporate alternative reboiler or reflux ratios. Potential changes to noncontinuous operations include adding an intermediate hold temperature above the current procedure to reduce long heatup or cooldown times, and using multiple receivers or quick pumpout procedures.

Objectives often can be achieved through upgrading of control elements — such as putting a manually-operated, or inadequately controlled, feed valve under continuous feedback and cascaded control. Instrumentation upgrades include the installation of an on-line sampler that quickly identifies targeted stream compositions and deviations from them. Equipment may be improved, for example, by altering the system pressure, supplying the existing reboiler with a higher-temperature utility source and the existing condenser with a lower-temperature coolant, or increasing heat-exchange surface areas by the addition of in-parallel or in-series heat exchangers, depending upon desired flow rates, temperature profiles, and available utilities.

A hypothesis-based problem-solving approach incorporating process simulation and field data is most effective for accomplishing the revamping objectives with minimal capital expenditures.

Use computer simulations — wisely

Computer simulators are readily available and are computationally accurate — making it easy to quickly simulate the full range of potential operating conditions. The majority of simulators employ an equilibrium-stage approach, based on vapor/liquid equilibrium (VLE) data, and model steady-state operations. Batch and dynamic simulators also are available. Process simulation packages are offered by, among others, Aspen, Chemstations, Hyprotech, and Simulation Sciences.

The key to an accurate simulation, especially with respect to the number of equilibrium stages, is accurate VLE data. Most commercially available simulators come with libraries of VLE data. Look also for in-house VLE data that may have been generated with previous distillation designs for the components within the system. Literature sources for VLE data include AIChE's Design Institute for Physical Property Data *(11)*, DECHEMA's Chemistry Data Series: Vapor-Liquid Equilibrium Collection *(12)*, and publications by Hala and coworkers *(13,14)*. VLE data also can be measured for the components under investigation and obtained from contract research laboratories for a fee. As a last resort, if actual VLE data are not available and measurement is impractical, a predictive model such as the UNIFAC group contribution method and its extensions (Sherwood and coworkers *(15)*) can be used, but only with extreme caution. Carlson *(16)* provides a decision-based approach for evaluating physical-property parameters for successful process simulations.

Despite the precision of simulators in calculating the number of equilibrium stages, translating theoretical to real stages remains an inexact predictive art based on empirical correlations such as the Murphree tray efficiency *(17)*. Further, each internals type, size, and configuration exhibits different hydrodynamics, and, until detailed understanding of the hydrodynamics is developed, empiricism will continue to predominate in the calculation or assignment of stage efficiencies. Once an internals type is selected, you are well advised to work closely with the internals vendor to determine the achievable stage efficiencies for a given design. In practice, stage efficiencies frequently are assigned based on the operation of similar systems of components and internals.

Whatever methods of modeling are used, perform simple checks on the simulation output to see if the simulation reflects real-world operation:

- Assess the simulator results (output) against those obtained from a McCabe-Theile diagram.
- Compare the output against data from the original startup and the current operation.

More details on how to make sure that you have a realistic and reliable simulation are given by Kister *(18)*.

Using the process simulator, you should do a methodical sensitivity study covering the full range of feed and operating conditions and product specifications. This allows you: (1) to determine what is achievable in the existing equipment, and how the controls and the control algorithms may need to be modified to achieve the target conditions; and (2) to begin to determine what a revamped system would be capable of achieving.

HOW TO ASSESS ENVIRONMENTAL ISSUES

Process wastes and emissions can have a major economic impact on distillation systems. Therefore, any revamping strategy needs to assess environmental gains and losses. First, identify what is causing the solid or liquid waste components in the current system. Determine if there are air or water emissions from the system, and whether they are fugitive or contained within the process streams. Also, ascertain where the emissions are escaping — that is, into the vacuum system, the cooling water streams, through the piping flanges, or somewhere else.

The two predominant process-stream materials that cause the most environmental concern are: (1) "still bottoms," which often consist of tarry residues that frequently contain a multitude of molecular species and exhibit high viscosity at ambient temperatures; and (2) high-vapor-pressure materials (material losses), which may slip through condensers into vacuum systems or become fugitive emissions through leaks in flanges and other equipment connections.

Reduce the amount of still bottoms

Still-bottoms or tarry-material formation frequently is caused by byproduct formation in the reactor and, if conditions permit, in the still. Distillation systems used to process effluents from more than one production line often are particularly susceptible to tar-forming reactions. An additional problem with still bottoms involves disposal. The presence of such high-molecular-weight products in the waste often results in additional waste, as processors try to manage their flow by: (1) controlling the discharge composition to include lighter components, such as process solvent; or (2) keeping the flow high or the outlet orifice larger than necessary to avoid any chance of plugging. Losses also occur when operators handle the high-viscosity material by diluting it with valuable solvents or raw materials.

For distillation yield maximization and waste minimization, it is necessary to obtain as much knowledge as possible about any potential (ongoing) chemistry occurring in the still or upstream that leads to tarry materials and their composition in the distillation feed. Because there may be no documented research or history of the still's chemistry, the only alternative may be to pinpoint as closely as possible the chemistry of the tar formation and its location. Pay particular attention to additional chemistry that may be occurring in multicomponent

mixtures. If your expertise in chemistry isn't up to the task, consult a competent chemist. Once you've determined the cause of tar formation, methods for avoiding it can be developed. Here are several options for reducing tar formation, ranging from economical to costly:

1. If tar forms because of components that originate upstream, look at the upstream process to determine how it can be changed. Address what is causing byproduct formation (yield losses) in the reactor, and determine if higher-purity raw materials can minimize or even eliminate it. Can the process be operated at different temperature or pressure conditions to avoid byproducts? If so, can this be accomplished with minor equipment modifications? Can the optimum residence time be controlled more tightly? Can the solvent system be changed? If the process is operating away from its optimal conditions, is this due to inadequate instrumentation and process control? Do detrimental nonuniformities, such as temperature or concentration gradients in the reactor due to heat-transfer mechanisms or mixing, respectively, exist?

2. If the problem is occurring within the distillation system, determine what is causing it. Can operating at different pressure or temperature conditions help avoid tar formation or concentration? Lowering the pressure may improve distillation recovery or reduce equipment-induced pressure drops such as vapor line restrictions. If the heat-transfer mechanisms are prompting thermal degradation or undesirable reactions, the reboiler can be redesigned, or different heat-transfer media can be utilized. If heat losses on certain surfaces are causing undesirable effects, the insulation can be improved. Evaluate whether these solutions could be accomplished with minor equipment modifications, such as more-effective condensers, improved heating or temperature control of the feed, or a retrofit of the column internals. Check to see if inadequate instrumentation and process control could be causing the tar formation.

3. Can recycling a stream from the distillation system to the upstream process cut tar formation? This also may offer the benefit of returning unconverted raw materials or perhaps providing a solvent source.

4. Can the distillation system produce other, marketable cuts that can reduce still bottoms? For example, adipic, glutaric and succinic acids from nylon-intermediate-manufacturing stills and crystallizers are sold as an additive in flue-gas scrubbers.

5. After achieving possible source reductions, you must reclaim or dispose of the remaining tar stream. Reclamation typically involves relatively expensive unit operations such as extraction or high-vacuum wiped-film evaporators and, so, often is cost prohibitive. Disposal options include off-site incineration and disposal in landfills — which are both costly and politically incorrect. Besides the expenses already incurred in raw-material procurement and upstream processing, landfilling or incineration runs from $0.15–1.50/lb of waste. And, the approval and permitting processes and the effort associated with the paperwork impose additional costs.

6. A more economic disposal option is to cofire tar in an existing boiler. This, however, may also mean consuming lighter components such as process solvents to maintain the flowability of the tar. It also mandates implementing a reliable firing configuration that won't plug, and ensuring acceptance of the system by the boiler operators. Last but not least, in most states, cofiring requires revisions to the facility's Clean Air Act Title V operating permit, which, depending upon the state's regulations, may take more than a year.

7. An operational technique for waste minimization with tars is to concentrate the tars, removing valuable components and, thereby, purging a lower quantity of waste. This frequently is achievable and effective in batch and semicontinuous distillations.

8. Startup and shutdown operations also must be assessed for waste generation, as practices may involve purging of the still contents, resulting in additional waste.

Run laboratory tests

The next step is to perform a laboratory distillation, preferably in a glass system to allow visual observations. Though this step is an uncommon one, it can provide valuable insights on the system at hand, helping the engineer to understand the chemistry that is occurring and to identify phenomena and their possible causes. By performing the distillation process under a similar temperature and pressure to plant conditions, it is possible to:

- observe any foaming effects;
- create conditions that might lead to plugging by solids or fouling by tars;
- detect sublimation of unknown components;
- spot a color change indicating that reactions are occurring;
- notice a change in viscosity;
- verify or discount any assumed chemistry (presuming samples can be taken); and
- characterize product streams, to confirm stream composition, especially impurities and byproducts.

HOW TO DEVELOP THE RIGHT STRATEGY

Armed with data from the previous steps, you now can develop the distillation-system-operation strategy that will create favorable conditions for yield maximization, operability, and waste minimization requirements. To do so, the following operational parameters must be specified: product stream composition, operability, onstream time, average column residence time, column holdup, and startup and shutdown procedures.

Conduct field trials

At this point, your knowledge level and revamping strategy should be at least 90% complete. Running tests now with the existing equipment should verify the strategy, while providing the final assessment of the existing equipment under the proposed conditions. Prior to the field trial, finish any remaining mass- and heat balances and system productivity-rate calculations. Throughout the field trial, the data logging should include a continuous assessment of how close the system is running to the target conditions; this is critical for determining what modifications are required to achieve desired operation. Hanson and coworkers provide a summary of preparation procedures prior to a distillation test run *(19)*.

The field trial should strive to simulate the full range and combinations of parameters including feed, liquid, vapor, boilup and reflux rates; temperature and pressure profiles; and the startup and shutdown procedures. If the revamping strategy involves a change of operational mode — for instance, from batch to semicontinuous operation — the trials may require extensive manual control to achieve the operational sequence. Limitations of the system should be identified and explicitly assessed.

The hydrodynamics, both liquid and vapor, of column internals should be evaluated at the projected typical, minimum, and maximum flow rates, and at the minimum and maximum achievable reflux rates. This requires that close attention is maintained to all system indicators such as temperature and pressure profiles, flows, and reboiler and condenser operation. Signs of liquid entrainment (jet flooding), liquid flooding, and liquid or vapor maldistribution — and corresponding losses in the separating efficiency — should be monitored constantly.

As the distillation progresses, get a compositional analysis of the feed, bottoms, overheads, and, if available, intermediate stage(s) within the column. This analysis will refine the mass balance calculations and computational modeling, and provide an assessment of the actual number of separation stages — that is, the stage efficiency.

The goal, throughout the field trial, is to clarify and confirm the three fundamental questions for the column revamp, specifically:

1. What are the required hydrodynamics to achieve efficient phase contact?

2. What are the number of stages necessary for the separation, based on the equilibrium thermodynamics of this distillation system?

3. What precautions, such as the avoidance of plugging, fouling, or the like, are needed for system reliability?

At the conclusion of the field trial, the revamping strategy should be nearing completion. You should have determined the operational mode and sequence, as well as conditions conducive for acceptable hydrodynamics, startup and shutdown procedures, and the instrumentation and the process control algorithm.

With this critical information, you now can turn to selection of the specific internals.

Start with the existing internals

Retrofitting better internals into an existing column can readily provide increases in capacity and fractionation capability. Such a revamp isn't always needed, however. At times, performance of existing internals can be improved significantly by understanding their inherent limitations and modifying the operation to overcome these limitations.

No one internals type, be it trays, structured packing, or random packing, is superior to the others in all applications; indeed, all can be made to fractionate the same chemical systems. Therefore, it behooves the revamping engineer to understand what the existing internals configuration can and can't accomplish before opting to retrofit a new set of internals.

There are, as we'll discuss, some general differences in performance between trays and packings, and these can serve as a guide for preliminary selection. The final comparison, however, should be between optimum trayed-column and packed-column designs. The optimal tray design is one that balances bubble and downcomer areas so that neither becomes a capacity pinch. Such a design then will maximize the fractionation efficiency and column capacity by specification of weir height and geometry, downcomer clearance, and the fractional hole area. Similarly, an optimal packed-column design is one that properly incorporates liquid and vapor distributors, packing type and size, and supports to avoid or minimize adverse liquid and vapor maldistribution.

Choose the right internal

The goal in selecting internals is to come up with a configuration that maintains effective vapor/liquid contact over the desired operating conditions, while minimizing pressure drop, liquid entrainment, weeping, corrosion, and fouling or plugging. At this point, it should be clear that column capacity is largely determined by column diameter, specifically its ability to handle the desired liquid and vapor flow rates without inducing adverse flooding. Groups, including Fractionation Research, Inc. (Stillwater, OK), and the Separations Research Program at the University of Texas at Austin, have performed fundamental research on system parameters that affect internals selection.

In general, most columns less than 36 in. dia. tend to favor packing, either structured or random. Beyond the general 36-in.-dia. division, the dimensionless flow parameter, FP, can aid in selecting column internals type. The flow parameter is the ratio of the square roots of the kinetic energy of the liquid to the kinetic energy of the vapor — that is, in mass units:

$$FP = (L/V)(\rho_V/\rho_L)^{1/2} \quad (1)$$

For optimized, modern 24-in.-spaced trays, 2–2½-in.-nominal-dia. packing,

and 67-ft²/ft³ structured packing, capacities and transfer efficiencies were evaluated. At a *FP* between 0.02 and 0.1, the structured packing exhibited approximately 50% higher efficiency; as the *FP* rises above 0.1, however, the efficiency advantage of structured packing rapidly declines with increasing *FP*. For details, see Ref. 20. In general, at a lower *FP*, for instance, less than 0.1, packing usually is the first choice for evaluation, while at higher *FP*, say, 0.2–0.3 and greater, trays generally are favored; between 0.1 and 0.2, there is no clear initial choice between trays and packing.

While a parameter like *FP* can help guide you to an initial choice, the subsequent internals design needs to be fully evaluated based on complete system information. If a significant concern exists, then assess another option.

Distillation trays

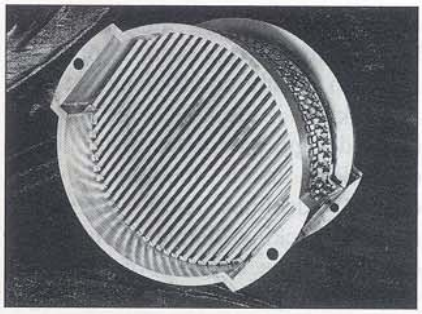
In the past, three basic tray designs — bubble cap, sieve, and valve — were the dominant distillation internals in the CPI. Bubble-cap trays assure total contacting in high-vapor/low-liquid contacting situations; however, such trays tend to be more expensive, heavier, and to produce higher vapor pressure drop, which leads to greater energy consumption. Sieve trays have less hardware and, so, are much lower in cost; they, however, offer limited turndown relative to bubble-cap or valve trays. Valve trays, which are intermediate in the price range, generally afford a larger operating range than sieve trays and also minimize weeping at low vapor rates and deflect entrainment at higher vapor rates. Valve tray designs often are proprietary and, therefore, close contact with the internals vendor may be required in evaluating possible configurations. Although all three types are still widely in use, improved tray designs recently have been developed, and should be considered first for an internals revamp.

The new high-performance trays enable distillation at higher capacities and efficiencies. When an existing column diameter is sufficient for the desired capacities but the trays are outdated, revamping via a straightforward tray replacement, optimized at the current vapor and liquid loadings, often is attractive. Vendors offering high performance trays include Koch Engineering (which recently bought Glitsch), Norton, and Nutter Engineering/UOP. Examples of high-performance trays are shown in Figures 1–3.

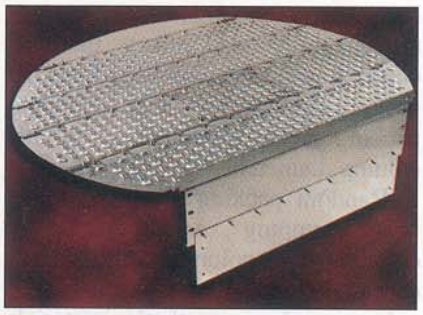
Random and structured packing

In columns where packing is an option, the installation of higher efficiency or higher loading packing often realizes significant increases in fractionation ability and capacity, respectively. Properly applied random and structured packing operate with considerably lower pressure drop (one-third to one-fifth less per theoretical stage) than correspondingly sized tray systems. Therefore, packing usually is selected over trays in applications ranging from atmospheric to low-pressure/high-vacuum. In general, operating at a lower system pressure and pressure drop raises the relative volatility, allows for lower reboiler temperatures, and may enable a reduced reflux ratio or a higher product purity. This also translates into lower utility usages. Packing, as already noted, also is favored for small-dia. columns, where fabrication costs for trays can be relatively high, and, to a lesser extent, in high-pressure distillations where tray columns are downcomer limited.

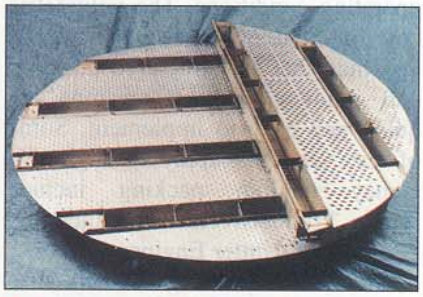
Random packing — This originated ages ago with crushed rock and glass, and progressed with the introduction of Pall rings in the 1960s. Today, a wide variety of random packing types are commercially available, with sizes ranging from ½–3½-in. nominal dia. Random packing has complex surfaces and high open area to minimize pressure drops while maximizing vapor/liquid contacting. Figure 4 shows an assortment of modern random-packing geometries.



■ *Figure 1. CoFlo high-performance tray. (Courtesy of Jaeger Products, Inc.)*



■ *Figure 2. Bi-FRAC high-performance tray. (Courtesy of Koch Engineering Co.)*



■ *Figure 3. VGMD high-performance tray. (Courtesy of Nutter Engineering/ UOP Inc.)*

If corrosion of the existing internals has been detected, consider a packing with different material of construction. Packing is readily available in various metals including specialty alloys, ceramics, plastics, and glass-reinforced plastics. Because of increasing understanding of the importance of liquid adherence to the packing surfaces, many vendors are modifying surfaces to increase wetability. Maintaining the physical integrity of packing (durability) also is important, as too often breakage

occurs — leading to fluid maldistribution, unwanted pressure drops, and losses in separation efficiency.

Packing size (diameter) is a significant design parameter. The height equivalent to a theoretical plate (HETP) declines with decreasing nominal packing diameter; this, however, is at the expense of increased pressure drop across the same height of column. While smaller sizes enable better mass transfer, they also can create higher pressure drop and lower capacity. This must be balanced against wall effects that can occur. A rule of thumb is to limit the packing diameter so that at least eight packing pieces would fit across the column diameter.

Random packing is most applied in revamping small diameter columns, large columns with noncritical product-stream specifications, and columns where synthetic materials are preferred, for instance, because of highly corrosive or low-temperature operating conditions. The volumetric cost for random packing typically is less than that for structured packing, and random packing revamps are quick and easy to implement. Some of the vendors supplying high-efficiency random packing include Jaeger Products, Koch Engineering, Norton, and Nutter Engineering.

Structured packing —This frequently is the preferred internal in revamps where high efficiencies or low pressure drop and high capacity are advantageous. Structured packing, which typically is fabricated from corrugated sheet metal or synthetic materials, offers an even-lower pressure drop per HETP and more capacity than random packing. Structured packing HETPs continue to decrease, thereby boosting its attractiveness for revamps where greater fractionation is a key objective. Because of increasing competition in the marketplace, the cost of structured packing has declined to the point where it effectively competes with random packing on price. Figure 5 shows an example of structured packing. Vendors include Jaeger Products, Koch Engineering, Norton, and Nutter Engineering.

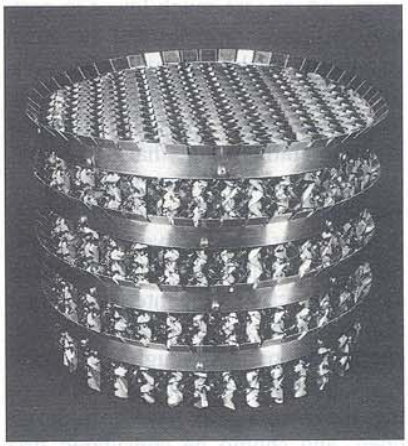
■ *Figure 4. An assortment of random packings. (Courtesy of Koch Engineering Co.)*

Liquid distributors — Properly designed and operated liquid distributors are critical in packed columns, because poor distribution will severely reduce vapor/liquid mass transfer. Liquid redistribution within a packed column also is crucial for effective mass transfer throughout the column height. In general, liquid redistribution plates should be inserted for every 8–10 ft of column height. Strigle *(6)* provides guidelines on liquid distributor designs.

Determine stage efficiency and required fractionation

Now, you are ready to evaluate the sufficiency of the existing column height and its corresponding number of stages. By coupling the field trial with the simulations, you should be able to verify the critical column parameter — stage efficiency. Internals vendors frequently can provide stage efficiencies for common systems. Although reliable stage-efficiency values may not be readily available for many revamps, the stage efficiencies can be calculated explicitly by comparing the simulations with the field results, which is, in effect, the conversion between theoretical and actual stages.

In working with the desired ranges of feed and all product streams, the required fractionation stages must be determined. The first option is to balance the number of stages against reflux ratio. By increasing reflux ratio, you boost the fractionation. To accommodate this change, the thermal capacity of the column (specifically, the reboiler and condenser) must be



■ *Figure 5. An example of structured packing. (Courtesy of Jaeger Products, Inc.)*

capable of handling the corresponding loads. Conversely, increasing the number of stages by adding height to the column (or revamping with higher efficiency internals) allows a lower reflux ratio and boilup rate for the same degree of fractionation and column productivity.

An increase in column height sometimes can be accomplished with an extension of a flanged vessel; a structural analysis of the column is required to determine if this is feasible.

HOW TO ANALYZE THE ECONOMIC IMPACT

Using the guidelines and the order of preferences described above, you can develop a strategy based on the economic realities of achieving customer requirements, system operability, and reliability. Now, you have to assess the strategy's costs, which primarily consist of:

- distillation system downtime;
- capital expenditures; and
- engineering requirements or fees.

In most revamp projects, downtime is the largest economic factor. Significant downtime can lead to lost market share or the need to buy from competitors to supply customer accounts. Further, in an integrated facility, downtime within the distillation system often can cause outages that negatively impact other units.

Capital expenditures usually are secondary to downtime costs. To estimate the cost for retrofitting column internals, consult the recent article by Bravo *(21)*. Investment for revamping the instrumentation and controls, ancillary equipment, such as the reboiler and condenser, and the column vessel can be estimated using standard techniques. When considering capital expenditures, always keep in mind that incorporating the proper system component to accomplish the strategy is more important than saving a few dollars on a lesser piece of equipment. Also, remember that one of the best ways to avoid unnecessary expenses is to resist the temptation to buy new equipment to solve every problem.

The third cost consideration involves the engineering requirements or fees to determine the best revamping strategy and design. It is most economical to handle the project internally at the plant level or to hire a distillation operations specialist. If, instead, you choose an outside engineering firm, bear in mind that some less technically proficient engineering firms operate on the same basis as equipment vendors — effectively charging a commission based on the size of the total project.

To reiterate: though it may cost more initially to take the step-by-step approach outlined here, this approach will truly identify what is needed to achieve the desired results. Ultimately, this process often yields much higher returns, especially from decreased downtime and eliminating unnecessary changes. The potential economic gains achievable through this approach include:

- increased system throughput;
- higher recovery of raw materials;
- better product yield;
- more efficient energy usage;
- reduced waste-disposal quantities and costs; and
- more efficient use of operating personnel.

PROVEN IN PRACTICE

I have applied this approach to a wide range of distillation processes. Here are some examples to provide a sense of what can be accomplished.

Solvent recovery

Company X decided to take a fresh look at one of its well-established, profitable manufacturing processes. Although the initial goal was to produce a higher-purity product at higher capacity, the project rapidly developed additional objectives. The plant manager was concerned about raw material costs and customer complaints about product purity and appearance. The production engineer was troubled that yields sometimes were low, the quantity of waste material was inconsistent, and that the waste material at times was difficult to handle, which affected the cost of disposal. From an environmental and safety standpoint, the process material was an EPA-listed air pollutant and considered a carcinogen. Ultimately, the issues at hand had led to a profitability limitation, which the company naturally wanted to overcome.

The chemists and engineers associated with the product line had identified at least 12 potential process causes of inadequate product purity and appearance. A field investigation was performed to ascertain when the product-quality and yield concerns became most prevalent. Both subsequently were correlated to the proportion of recycled feed materials from the distillation system to the reaction and separation trains.

The existing distillation system processed reaction and separation solvents and other reactants, and much solvent was being lost with the waste materials. Also, higher volatility byproducts were being formed both in the reactor and the still itself, which led to increased disposal costs and potential liability from shipping large quantities of hazardous waste on a daily basis.

The system, which had been in operation for more than a decade, consisted of a single-stage column (with no column internals) fitted with a jacketed, steam-heated kettle reboiler. The vacuum system was piped to a single receiver with two condensers in series. It was a batch system with minimal process controls, mainly on/off valving and a few temperature and pressure gages.

One of the previously proposed engineering designs was to achieve greater fractionation of three components via a column internals revamp and extensive process instrumentation and control. The components, in order of decreasing volatility, were the reaction solvent, the separation solvent, and the reaction byproducts. To reduce the solvent losses through the condenser and into the wastewater system, the design replaced the

existing steam-jet vacuum with a vacuum pump system having a refrigerant-based condenser system. This proposed revamp would have cost in excess of $2,000,000. It, however, would not have solved the product quality issue, improved the environmental aspects of the process, or reduced the disposal costs.

Through careful analysis using the approach discussed in this article, the revamping engineers came up with a far better alternative.

By identifying what the problems were upstream, the engineers more realistically determined what would make the most sense in the distillation process. The challenges to the distillation included wide variations in the feed to the still due to inconsistencies among reaction endpoints. This became the focus of a short-term process-development project. It led to alteration in the reaction conditions and conversion of the reaction and separation process from a three component system to a two component one employing the same lower-volatility solvent for both the reaction and separation.

Laboratory distillation determined that the reaction byproducts were extremely viscous, reaching more than 1,000 cP at room temperature, and that a stagnant reboiler mixture exhibited detrimental foaming characteristics. In the laboratory, the solvent in the still bottoms could be limited to a few percent. In the plant, the properties of the still bottoms, including viscosity and solvent holdup, varied widely even though the same temperature and vacuum endpoints were consistently used. The laboratory distillation proved that the single-blade propeller agitator in the still simply didn't provide agitation and, therefore, the interfacial transfer quickly declined as the surface became quiescent in the early stages of solvent removal. So, the kettle was retrofitted with a dual axial impeller, which induced a high-velocity axial flow and more interfacial transfer area.

By using the single solvent, the distillation was converted from a batch operation to a semicontinuous one, which achieved several objectives for the revamp. The first was increased solvent recovery, which enabled the plant to process considerably higher quantities. Final product purity also was greatly improved due to better purity of the solvent. Additionally, because the distillation bottoms' concentration and viscosity were controllable, the company was able to pump the bottoms to the boiler house for reliable on-site destruction, which was significantly less expensive (and more politically correct) than shipping it off-site.

In summary, a costly revamp was avoided by understanding the limitations of the existing distillation system, modifying the upstream process, converting the operational mode, addressing the environmental concerns, and then retrofitting only a minimal amount of equipment.

Pharmaceutical intermediate purification

Company Y needed to revamp a batch distillation operation to meet customer requirements for purity of a particular pharmaceutical intermediate. The existing column contained structured packing with four receivers in parallel and had variable reflux control based on single-loop controllers. It was operated under various vacuum settings utilizing a two-stage steam-jet system. The process was very operator intensive, requiring up to 80% of the operators' available attention. Additionally, because product specifications were not met, large quantities had to be reprocessed.

The challenges included variable feed concentrations from one batch to the next. Also, the operation produced six components, and the purity of the salable one — a component of intermediate volatility — was very inconsistent, typically ranging from 90–95%, while the customer demanded purity levels in the >95% range. Unacceptable cross-contamination of the lightest components into the salable component also was occurring, and the true distillation yields were unknown.

To achieve the desired distillate purity, the design engineers analyzed the system and developed two revamping proposals. One involved replacing the batch still with a thin film evaporator and a three-column distillation train including condensers, reboilers, accumulators, pumps, and controls. The second design retained the existing still, followed by a three-column distillation train and its associated equipment and controls. Either design would have required a capital expenditure in excess of $1,000,000.

Fortunately, the approach discussed in this article then was used. On-site analysis identified several problems within the existing operation. One of the key trouble areas was that liquid maldistribution frequently occurred throughout the batch distillation process, impeding fractionation. The existing column was operated on a variable reflux algorithm; the equipment, however, had been oversized to the point that adequate liquid distribution was maintained only at higher reflux rates. The control algorithm and the structured packing internals, coupled with the batch operation mode, also restricted the productivity in the larger fraction cuts (the five distillation cuts ranged from a few percent to 45% of the feed); operators frequently overrode the system controls to improve boilup rates. The feed composition to the still varied considerably — however, most of the variance could be attributed to two components, one of which was the highest volatility component.

After the field observations were made, the system was modeled on a simulator — first, batchwise and, then, in a semicontinuous mode, adjusting the reflux ratio to maintain adequate internal liquid rates for the existing column internals. Through the simulations, the revamping engineers determined that the current

equipment could provide the desired fractionation with the desired purity, improved yield, and at higher productivity rates, if the system was converted from batch to semicontinuous operation.

Next, the revamping strategy was to be confirmed in the field. The installed instrumentation and the control, however, were not adequate for semicontinuous operation; so, the field trial was conducted almost exclusively using manual control. During the plant trial, successful fractionation was achieved for all six fractions, both with respect to distillate purity and cross-contamination. Consistent increases in product purity and a gain in product yield also were attained.

Ultimately, the revamp did not require additional columns, but only a change from batch to semicontinuous operation, as well as modified programming of the controllers. As a result, more than $1,000,000 of unnecessary capital expenditures were avoided, along with the costly downtime to implement them. Further, the required operator involvement was reduced, and product rework was virtually eliminated. There also was an environmental benefit to the revamp, in that waste disposal was dramatically decreased due to greater productivity per heel discharge.

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Achieve the optimum

The approach presented here will enable you to effectively develop a practical and profitable revamping strategy for distillation systems throughout the CPI. It is not intended to be a "cookbook" solution; instead, it is more of an algorithm that should be tailored to the specific situation. Always remember that coming up with the most economic overall solution must involve taking into account reliability, operability, downtime, and capital investment. Although intuition does play a role, it is developed only through practical experience and a thorough understanding of the engineering process. The factors to consider in any distillation column revamp include:

- the upstream process(es);
- waste minimization;
- instrumentation and process control;
- plant and laboratory data;
- process simulation; and
- the chemistry required for yield maximization.

As I have highlighted, using this approach can pay big rewards. CEP

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