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A BASIC PRIMER ON LIQUID-LIQUID EXTRACTION

An introduction to LLE and agitated LLE columns | By Don Glatz and Brendan Cross, Koch Modular

Chemical engineers are often faced with the task to design challenging separation processes for product recovery or purification. This article looks at the basics of one powerful and yet overlooked separation technique: liquid-liquid extraction. There are other unit operations used to separate compounds, such as distillation, which is taught extensively in chemical engineering curriculums. If a separation is feasible by distillation and is economical, there is no reason to consider liquid-liquid extraction (LLE). However, distillation may not be a feasible solution for a number of reasons, such as:

- If it requires a complex process sequence (several distillation columns)
- If the components to be removed or recovered have low volatility
- If it results in high CapEx and OpEx
- In these instances, LLE may be the best separation technology to pursue.

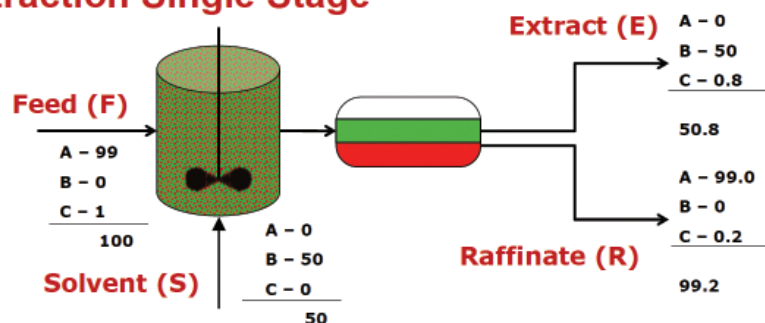
The basics of liquid-liquid extraction

While distillation drives the separation of chemicals based upon differences in relative volatility, LLE is a separation technology that exploits the differences in the relative solubilities of compounds in two immiscible liquids. Typically, one liquid is aqueous, and the other liquid is an organic compound.

Used in multiple industries including chemical, pharmaceutical, petrochemical, biobased chemicals and flavor and fragrances, this approach takes careful process design by experienced chemical engineers and scientists. In many cases, LLE is the best choice as a separation technology and well worth searching for a qualified team to assist in its development and design.

Figure 1 can be used to gain an understanding of exactly what LLE is. A feed consisting of mostly component A plus component C (solute) is well mixed with solvent B. The goal is to achieve mass transfer of solute C from feed A into solvent B. After the maximum amount of mass transfer is achieved, the phases

Simple Extraction Single Stage



Fraction Unextracted $\rightarrow U = \frac{\text{Solute in Raffinate}}{\text{Solute in Feed}} = \frac{0.2}{1.0} = 0.2$

Distribution Coefficient $\rightarrow M = \frac{\text{Conc. Solute in Extract}}{\text{Conc. Solute in Raffinate}} = \frac{0.8/50}{0.2/99} = 7.92$

Extraction Factor $\rightarrow E = \left(\frac{S}{F}\right)(M) = \left(\frac{50}{99}\right)(7.92) = 4.0$

Figure 1

Cross Flow Extraction

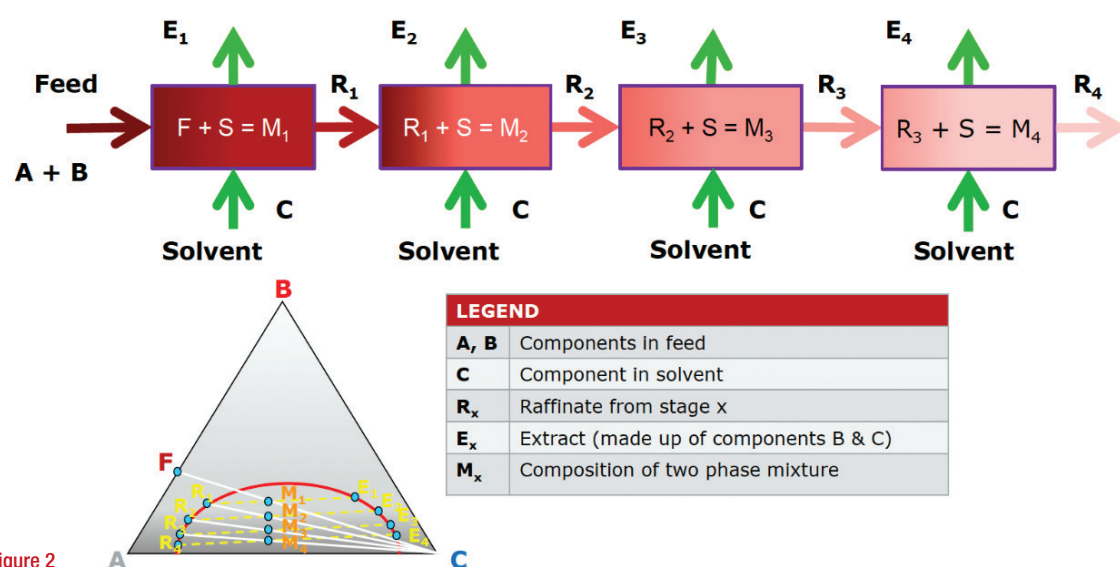


Figure 2

are separated into an extract phase (solvent C plus the extracted solute B) and a raffinate phase (feed A plus the residual solute C) and possibly the soluble amount of solvent B.

Assuming that equilibrium was achieved during the mixing phase, this process represents a single theoretical stage of extraction. As shown in Figure 1, for a single theoretical stage, there are a number of parameters identified at equilibrium, including fraction unextracted, distribution coefficient and the extraction factor. The key for LLE is to select a solvent with as high a distribution coefficient as possible for the solute to be extracted and as low as possible of all other components present in the feed.

For most LLE processes, a single stage of extraction such as this will not provide a sufficient amount of solute removal from the feed stream. In order to achieve higher levels of extraction, there are two flow patterns in which LLE can be operated: cross flow extraction and countercurrent extraction.

Cross flow extraction consists of a series of mixing and decants using fresh solvent each time. Thus, after the phase have separated, fresh solvent would be added to the raffinate and once again they would be mixed and allowed to decant. This procedure would be repeated as many times as necessary to get from the feed concentration of solute C to the required raffinate concentration, shown as R4 in Figure 2.

In countercurrent extraction, the feed flows in one direction from stage to stage while the solvent flows in the opposite direction relative to the feed. Both liquid phases are flowing continuously through the extractor. This type of process is shown for a column type contactor in Figure 3. In Figure 3, the feed is the heavy phase so it is added to the top of the column. It is also the continuous phase, so it essentially fills the column. The solvent is the light phase so it is added to the bottom of the column. It flows as droplets up through the continuous phase and thus it is the dispersed phase in the column. Rules of thumb for which phase should be continuous and which phase should be dispersed are provided later.

An important step in understanding an LLE process and eventually designing LLE columns is determining the liquid-liquid equilibrium data for the solvent of choice. The equilibrium data provides the equilibrium curve shown in Figure 4. The operating line in Figure 4 is determined by the actual inlet and outlet concentrations of solute during steady state column operation. These two lines allow for a graphical determination of the number of theoretical stages. The total number of theoretical stages required can be stepped off from the operating line to the equilibrium line, as shown in Figure 4, and is analogous to the method used for absorption and stripping columns.

As an example of this procedure, Figure 5

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Countercurrent Extraction

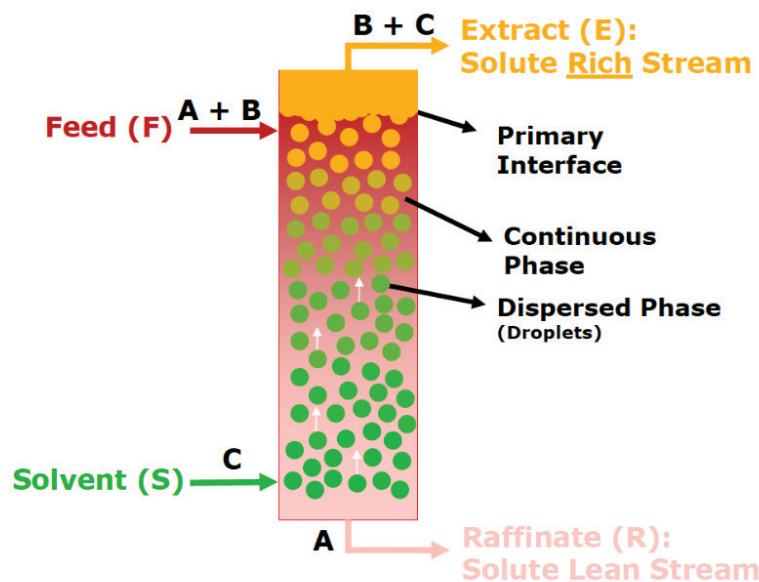


Figure 3

LLE Equilibrium and Operating Lines

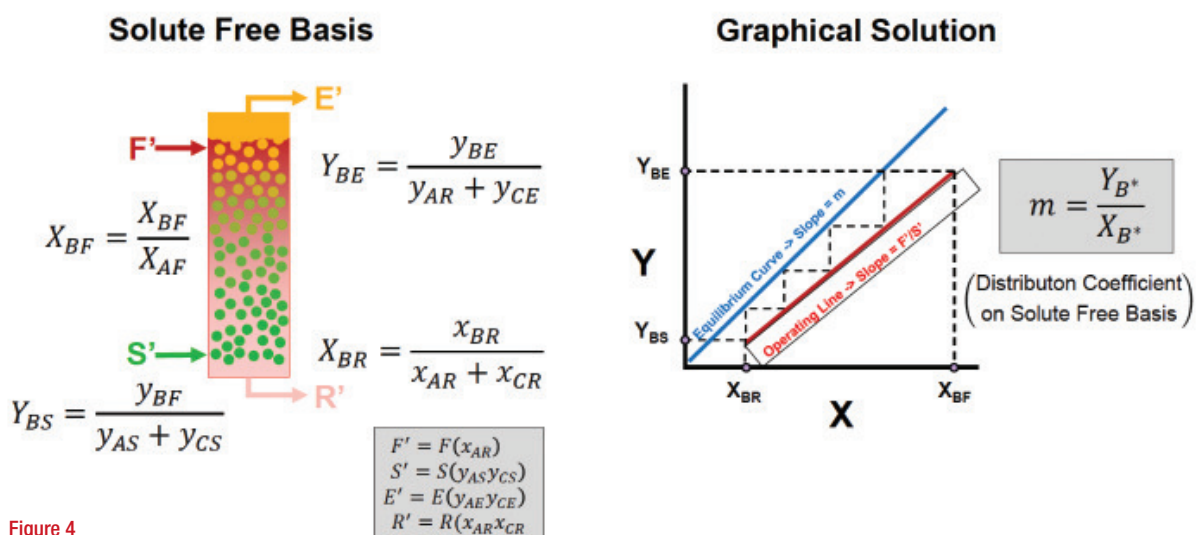


Figure 4

represents the extraction of a carboxylic acid from a fermentation broth using an organic solvent. The red line represents the equilibrium data generated using actual feed and solvent determined via laboratory testing. The operating line is drawn by determining the material balance required to achieve 95% recovery. In order to achieve 95% recovery of the carboxylic acid, three theoretical stages are required.

Another way to estimate the number of theoretical stages required for the LLE system is using the Kremser Equation. This equation considers the distribution coefficient, extraction factor and concentration of the solute in the feed, raffinate and solvent, all on a solute-free basis.

A graphical representation of the Kremser equation using different extraction factors is shown in Figure 6.

When is it recommended to use LLE?

- LLE is often used for the removal of high boiling components from dilute aqueous streams such as wastewater or fermentation broths. Two

great examples of this would be phenol extraction using MIBK and acetic acid extraction using ethyl acetate. Distillation being used as the sole separation technology for these applications would be cost-prohibitive because of the high amount of energy required. For example, if an aqueous feed contains < 30% acetic acid, liquid-liquid extraction is usually a more economical solution than distillation for removal and recovery of acetic acid because the operating costs associated with distilling away the water far outweigh the capital costs of the liquid-liquid extraction process equipment.

- LLE is also suited for the removal of hydrophilic particles from organic streams. For these applications, water is the solvent. A couple of examples of this type of application would be water extracting DMSO from a polymer solution and neutralizing & water extracting acids from organic streams.
- When recovery of non-volatile components is

Graphical Determination of Theoretical Stages

95% Solute Extraction, S/F = 1.0 mass basis

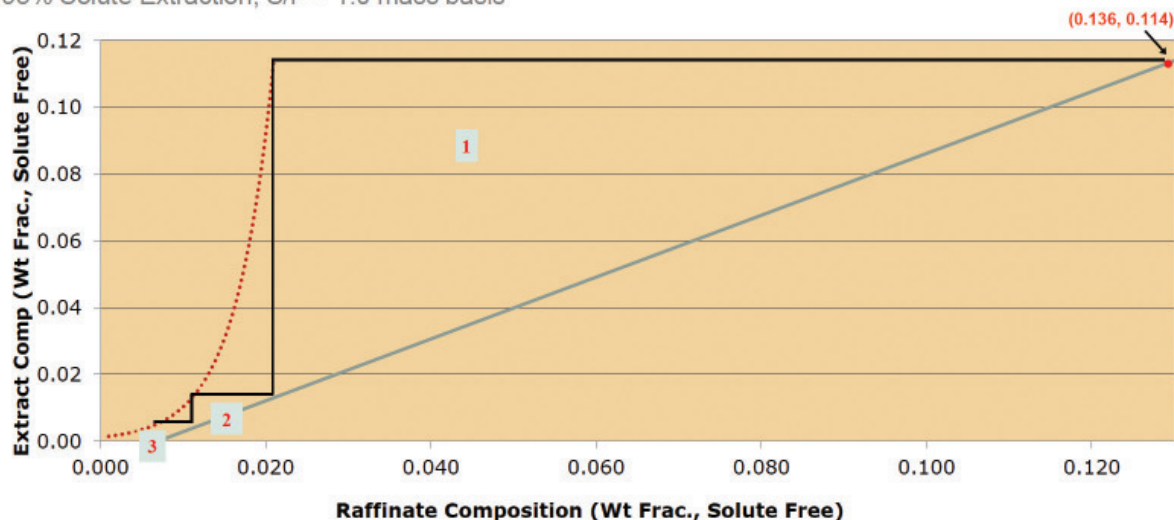


Figure 5

Kremser Equation

$$n = \frac{\text{LOG} \left[\left(\frac{x_f - y_s/m}{x_n - y_s/m} \right) \left(1 - \frac{1}{E} \right) + \frac{1}{E} \right]}{\text{LOG} E}$$

- n = Number of theoretical stages required
 x_f = Conc. of solute in feed on solute free basis
 x_n = Conc. of solute in raffinate on solute free basis
 y_s = Conc. of solute in solvent on solute free basis
 m = Distribution coefficient
 E = Extraction factor = $(m)(S/F)$

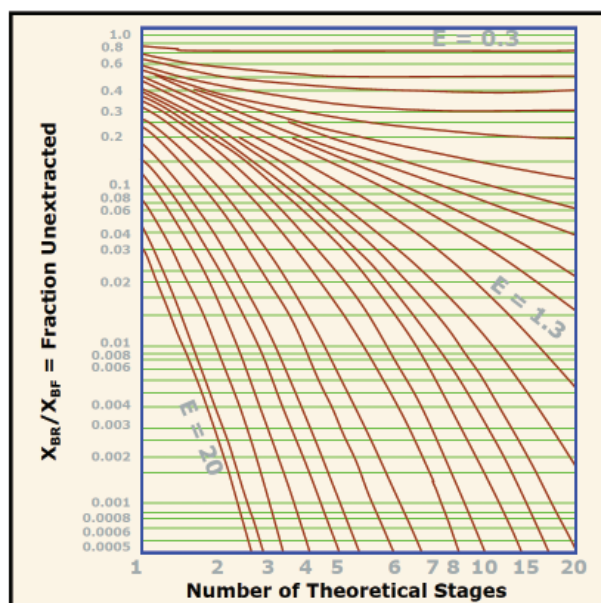


Figure 6

“The benefits of LLE position it as a powerful separation technology able to achieve product purification and recovery, particularly where distillation is not an economically viable option.”

required, LLE is an excellent choice, particularly as compared against distillation. Regular distillation is inefficient because of the high energy consumption that is required to boil water away from the non-volatiles. LLE is a better choice from an operating cost perspective. Extraction and recovery of metals are classically done via LLE processes.

- If the compound of interest is heat-sensitive and decomposes with temperature/time, then distillation may not be a good choice. LLE can often be performed at low temperatures at atmospheric pressure and thus prevent such decomposition.
- When the feed contains azeotropic mixtures, a complicated distillation process (such as a 2-column, pressure swing operation) will be required.

LLE can be considered as a cost-effective alternate if the proper solvent can be identified.

LLE column technology

As Figure 7 shows, there are three major types of extraction equipment: mixer settlers, column contactors, and centrifugal extractors. This article will specifically focus on LLE columns.

There are two major types of liquid-liquid extraction columns: static and agitated.

There are three types of static columns: spray towers, packed columns and trayed columns. Spray type columns are rarely used because they typically only provide up to one theoretical stage. Packed columns are used in refining and petrochemicals applications and use random or structured packing such as SMVP

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packing. Sieve tray columns are also used in the refining and petrochemicals industry. These columns consist of a series of sieve trays with either upcomers or downcomers, depending on which phase is continuous.

Agitated extraction columns offer a number of advantages over static columns. The primary advantage is that agitated columns offer many more theoretical stages than static column, thus the amount of solvent required is significantly reduced. The primary operating cost for an LLE process is often the downstream recovery of the solvent, usually by distillation. Thus, the operating cost for LLE systems using an agitated extraction column will result is a large reduction in the operating cost as compared to using a static column. For agitated columns there are three main types: pulsed, rotary and reciprocating.

Pulsed columns are often used in the nuclear and inorganic chemicals/metals industries. Examples of the internals use for pulsed columns include packing, sieve trays and disc and donut. For these columns, the internals are stationary, and agitation is applied by pulsing the contents of the column with a certain amplitude and frequency.

Columns with rotating internals include RDCs (rotating disc contactors) and SCHEIBEL columns (turbine type impellers). Based upon data published in the literature, the SCHEIBEL column has the highest efficiency. The KARR column is unique in that it utilizes a plate stack that reciprocates. This type of mixing action has been proven to be optimal for systems with low density difference between the phases and/or low interfacial tension — i.e. systems that tend to emulsify easily such as fermentation broths. RDC, SCHEIBEL and KARR columns are all used in the chemical, biochemical, petrochemical, refining, mining/metals and pharmaceutical industries.

Factors influencing the design of LLE columns
There are many factors that must be considered when looking to design an extraction column, including the following:

- Tendency to emulsify
- Effects of impurities or solids
- Coalescing/wetting characteristics

Major Types of Extraction Equipment

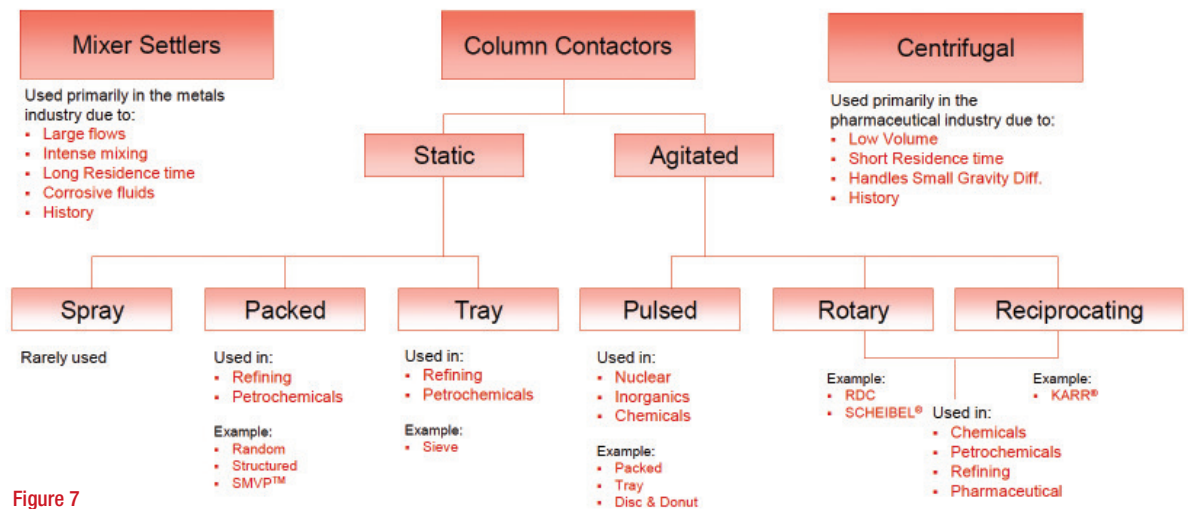


Figure 7

KARR® Reciprocating Column

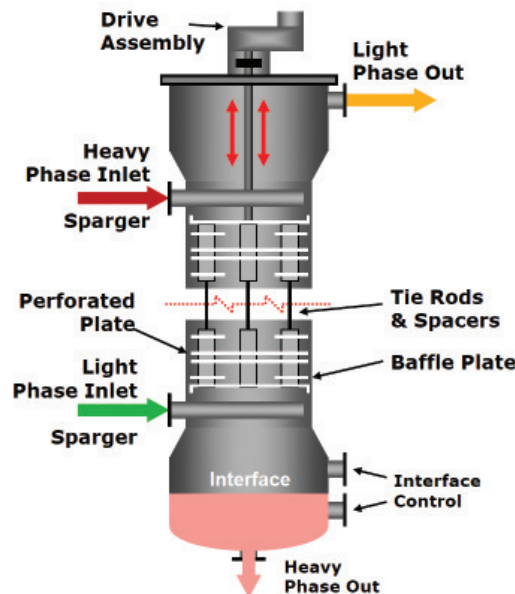


Figure 8

Characteristics

- Highest capacity:
 - 750-1,500 gal/ft²-hr
 - 30-60 M³/M²-hr
- Good efficiency
- Good turndown capability (4:1)
- Uniform shear mixing
- Best suited for systems that have slow phase separation or emulsify
- Optimal design for systems with suspended solids
- Typical commercial operating speed: 15 – 70 SPM

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- Phase separation time
- Mass transfer rate
- Rag layer and/or emulsion band formation
- Phase ratio variability and density gradients
- Axial and radial mixing
- Interfacial and drop turbulence effects

Trying to factor all of these variables into a theoretical design has proven to be very difficult. Since there are so many variables in play, the best method of design for liquid-liquid extraction columns is via a well-defined pilot test, followed by proven empirical scale-up. The purpose of pilot plant testing for LLE is to determine the optimal throughput capacity (which sets the diameter) and the HETS (which sets the height of the column). During pilot testing there are a number of variables that must be considered in order to provide the optimal column design. A brief description of these variables follows:

Selecting the continuous vs. the dispersed phase

Designing a column with the best choice of continuous and dispersed phases is critical for an extraction process. In the end, the pilot plant testing will determine the best mode of operation. However, some rules of thumb are as follows:

- **Phase flowrate ratio** — For static columns, the most efficient mode of operation is for the low flowing phase to be the continuous phase. However, for agitated columns the high flowing phase should be the continuous phase.
- **Viscosity** — If either the feed or solvent stream is very viscous, then this almost always is selected as the disperse phase in the column.
- **Surface Wetting** — It is important that the continuous phase preferentially “wets” the internals surfaces of the column. If the disperse phase wets the internals, then the droplets will coalesce resulting in loss of efficiency and throughput. As a rule of thumb, if the aqueous phase is the continuous phase, then the internals should be metal. If the organic phase is the continuous phase, then the internals should be PTFE or an analogous material.
- **Marangoni Effect** — The result from this phenomenon is that mass transfer of the solute from the continuous to the dispersed phase will be more efficient than transfer from disperse to continuous phase.

Other LLE phenomena

Interface behavior (rag layer, emulsion band) — Very often in LLE processes, either a rag layer (solids) or an emulsion band (2-phase region) forms at the interface. Pilot plant testing with actual feed and solvent can identify either one of these and the degree to which they are problematic, so that acceptable solutions can be identified.

Entrainment — Entrainment is not about soluble material, but rather when discrete, non-soluble droplets of one phase exit the column with the other phase. The degree of entrainment can be easily measured during pilot test operating by using a standard laboratory centrifuge. If the entrainment is too large, there are a number of corrective actions such as expanding the diameter of the disengaging chamber and putting a coalescing element in the disengaging chamber.

Flooding — When the dispersed phase stops flowing countercurrently to the continuous phase, this is considered flooding in an extraction column. It generally occurs when the continuous phase velocity gets too large (capacity limit) or in agitated columns when the agitation speed is too high, thus creating dispersed

“In many cases, LLE is the best choice as a separation technology and well worth searching for a qualified team to assist in its development and design.”



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phase droplets that are too small. It is easy to identify flooding during pilot plant testing in a glass shell column. In actuality, it is a good idea to determine the flood point during testing, and then to back off from those conditions (such as lowering the capacity and/or lowering the agitation speed) for finding the optimal operating conditions.

Pilot testing and LLE column scale-up: recovery of acids from fermentation broth

This was an application where an end-user was looking to recover 95% of the carboxylic acids from an aqueous fermentation broth feed stream. The fermentation broth contained roughly 5% carboxylic acids.

Ethyl acetate was selected as the solvent for this extraction process because of its relatively high distribution coefficient, but was known to emulsify easily when in contact with the fermentation broth in extraction columns that used radial agitation. This limited the operation of the extraction process during initial pilot testing, and also inhibited high levels of extraction.

Therefore, the KARR column was selected for this application because of the reciprocating action of the plate stack, which is designed specifically for systems that have a tendency to emulsify. Figure 8 shows the general characteristics of a KARR column.

First, pilot tests were performed for carboxylic acid with ethyl acetate. A 25-mm diameter glass KARR column with a 12-foot plate stack was used. The full set-up is shown in Figure 9.

The pilot tests demonstrated that the KARR Column was the correct choice for design, as the target extraction of 95% of carboxylic acids from the broth was achieved, and the solvent-to-feed ratio was reduced from the anticipated value of 2 to 1.5. The recovery at room temperature was between 96-97% and at 40-45°C, it increased to 98-99%.

The design capacity for the recovery of carboxylic acid was determined to be 650-700 GPH/ft² (26.5-28.5 m³/m²·hr) based upon feed and solvent rates from the successful pilot test.

Operating conditions from two select runs from the pilot test were used to design a commercial scale extraction column. See the highlighted runs in Table 1.

Pilot KARR® Column for Carboxylic Acids
1" diameter x 12' Plate Stack

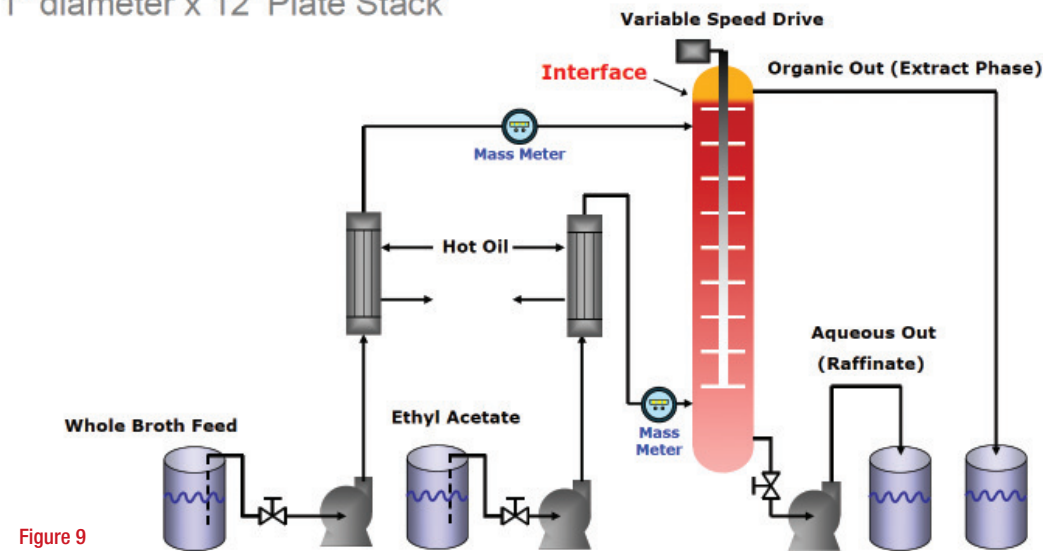


Figure 9

TABLE 1: Pilot Plant Data for Fermentation Broth Extraction						
All runs performed in a 25 mm diameter, KARR® Column						
RUN #	Plate Stack Ht. (feet)	Capacity GPH/ft ²	Temp. C	S/F Ratio	Agitation SPM	Acid Recovery %
1	12	700	23	1.7	30	95.0
2	12	700	23	1.7	40	97.5
3	12	700	23	1.7	50	Flooded
4	8	700	23	1.7	30	93.3
5	8	700	23	1.7	40	Flooded
6	10	700	23	1.7	30	96.3
7	12	650	23	1.5	30	96.5
8	12	500	23	1.5	40	97.6
9	12	700	23	1.5	30	94.3
10	12	650	45	1.5	30	98.3
11	12	650	45	1.5	50	98.7
12	12	650	45	1.5	60	Flooded

Table 1

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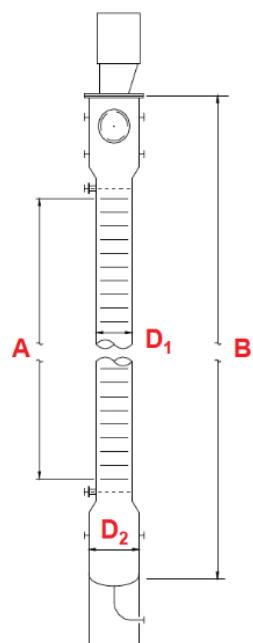
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- Expanded Ends
Diameter = 44" | 1.1M (D_2)
- Plate Stack = 38'-0" | 11.6M (A)
- Overall Height = 50'-6" | 15.4M (B)

Figure 10

The diameter of the commercial scale column was scaled up directly from the pilot test by applying the pilot test capacity to the commercial scale feed and solvent rates. This resulted in a diameter of 22 inches. For this process, the commercial scale feed and solvent rates were 12.4 GPM and 15.2 GPM, respectively.

The height of the plate stack (or agitated section) in the commercial column is a function of the pilot test column plate stack height, the pilot test column diameter and the commercial scale column diameter. Scaling up to the commercial scale extraction column resulted in an agitated section height (or plate stack height) of 38 feet.

The final design includes expanded ends (to inhibit entrainment) at 44-inch diameter and the overall height was calculated to be 50 feet, 6 inches (Figure 10).

In addition to the extraction column, downstream solvent recovery was provided using traditional distillation technology.

Conclusion

The benefits of LLE position it as a powerful separation technology able to achieve product purification and recovery, particularly where distillation is not an economically viable option. A successful, commercial-scale LLE process can be assured by completing a process development program involving laboratory work to generate liquid-liquid equilibrium data, pilot

plant testing to generate scale-up data and then using established design correlations to accurately design a LLE column. LLE technology is used in a variety of industries where thousands of extraction columns dot the chemical processing industry landscape. **PR**



Don Glatz is the Manager of Extraction Technology at Koch Modular. Don's activities include the evaluation and optimization of extraction processes plus scale-up and design of extraction systems. He has been working in this field for the past 30 years and has published a number of papers and articles covering this subject. Don holds a BS in chemical engineering from Rensselaer Polytechnic Institute and an MBA from Fairleigh Dickinson University.



Brendan Cross is a Senior Process Engineer at Koch Modular that works in the Extraction Technology Group. Brendan is responsible for liquid-liquid extraction application evaluation, extraction column and pilot test design, and commissioning and process startup. He has been with Koch Modular for over 10 years and also has experience in distillation and process development. Brendan holds a BS in chemical engineering Columbia University.

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