Mixing Equipment in Liquid-Liquid Extraction

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I. INTRODUCTION

Liquid-liquid extraction is a common industrial application. However, since additional separation steps must often be made, either by an additional liquid-liquid extraction step or by vapor-liquid separation, it is usually the last choice for separating two or more miscible liquids.

In addition to the usual solvent used in extractions, new techniques of utilizing liquid ion exchange materials have been increasingly used. This is especially true in the hydrometallurgy field where these selected ion exchange materials can preferentially extract certain ions from mixtures.

The market for liquid-liquid extraction apparatus is about one-fifth that of the market for gas-liquid contacting equipment. However, the number of new devices described and studied equals or exceeds that of vapor-liquid contacting equipment.

Rotating mixing impellers are used exclusively in gravity-type columns and mixer-settlers. There are three principal types of columns used commercially in the United States:

1. The rotating disk contactor [1-10], Fig. 1.
2. The Scheibel column [11, 12], Figs. 2 and 3.
3. The Oldshue-Rushton column [13], Fig. 4.

Several other columns have been described recently in the literature; references to these are cited later.

Mixer-settlers are very common. Lately, several types of pump-mix and box-type extractors have been used which have equipment features that can be used to advantage in particular applications (Fig. 5).

On a commercial scale, the manufacturing cost of countercurrent extractors often determines which of the several types is to be used.

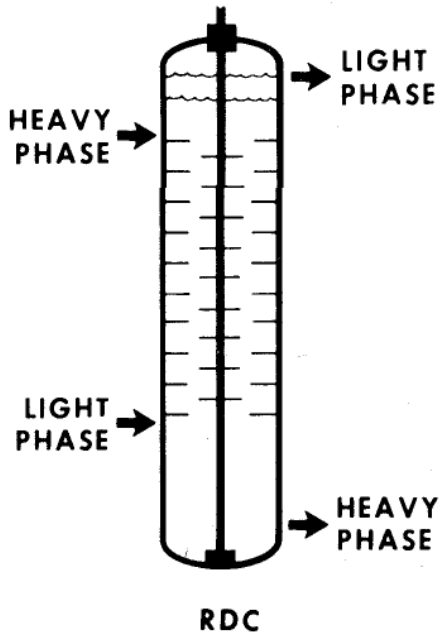

Fig. 1. Schematic of rotating disk contactor.

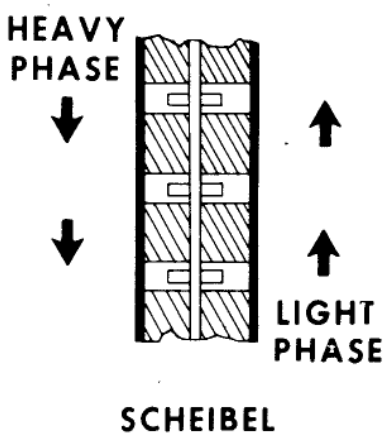

Fig. 2. Schematic of original Scheibel column.

Many applications require expensive alloys or plastic coatings, so that practicalities of construction and maintenance often outweigh any advantages claimed for more complex internal fittings, baffles, or calming zones.

The initial Scheibel columns used the concept of phase separation and coalescence between the mixing stages [11]. The recently modified Scheibel column [12], the rotating disk contactor [1-10], and the Oldshue-Rushton column [13] maintain a continuous dispersion with

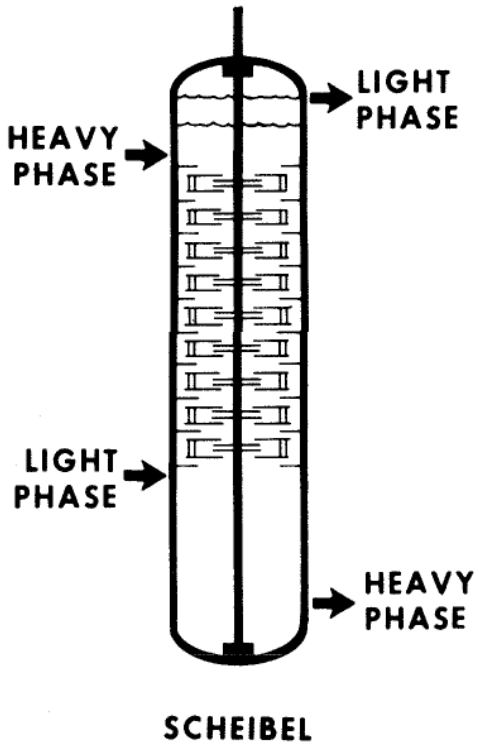

Fig. 3. Schematic of modified Scheibel column.

coalescence or settling in either top or bottom. The major question that arises when one compares these two concepts is whether a more intense agitation level in the mixing zone followed by phase separation in the calming zone is a more effective use of total column volume than a lower level of agitation with a continuous dispersion maintained throughout the entire system.

To date, the method more commonly used is continuous dispersion maintained in each of the stages with phase separation at only one end. Some recent columns (classified later) that have separate calming and settling zones have not yet been used commercially to any extent, and it remains to be seen whether the additional internals, especially in alloy construction, give a more economic column.

Many other considerations enter into the mixer design. The power levels in continuously dispersed extraction equipment are normally quite low, so that the cost of mixer and associated power is not a major item in the overall cost of a continuous countercurrent extraction column. There is usually some maximum mass transfer rate that can be obtained in a given volume of two-phase liquid systems while still allowing the

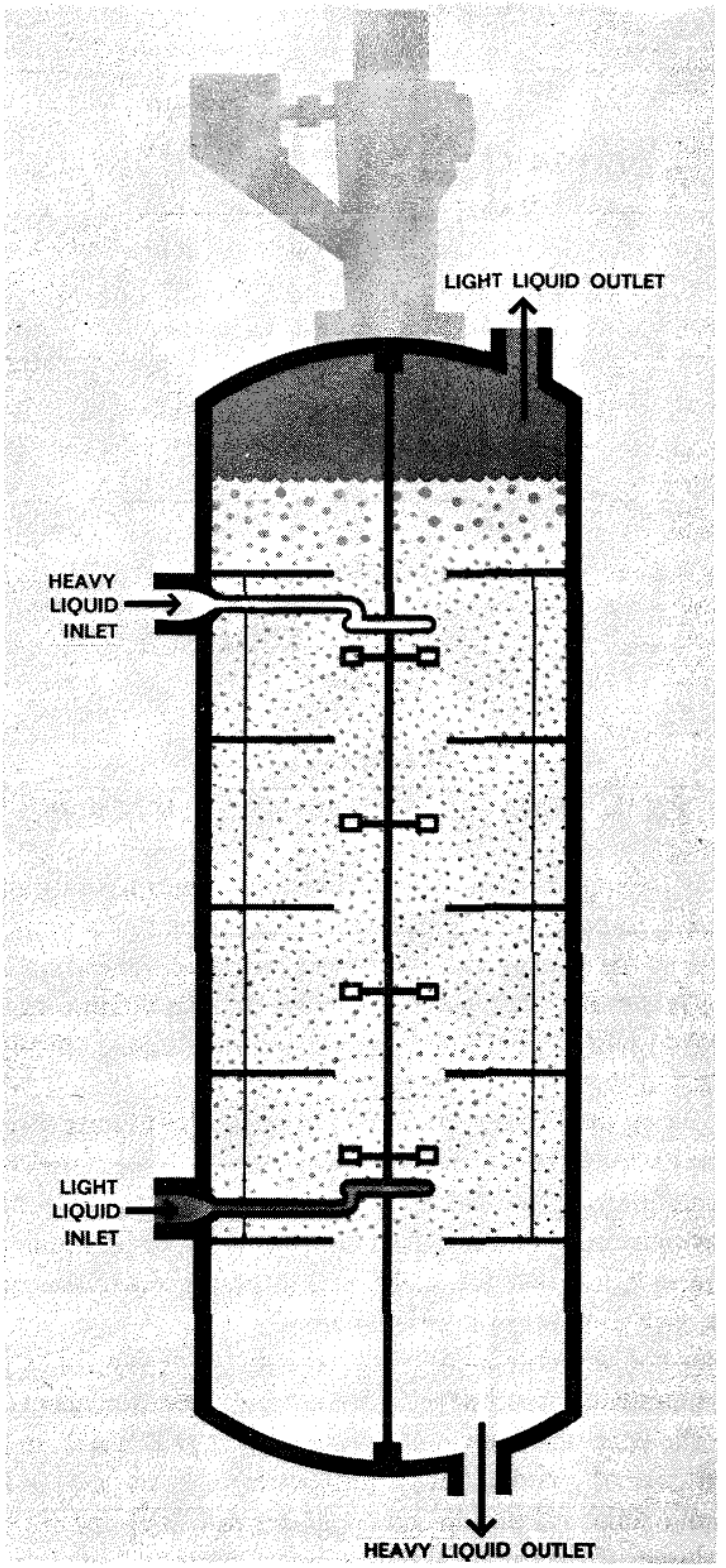

Fig. 4. Schematic of Oldshue-Rushton column.

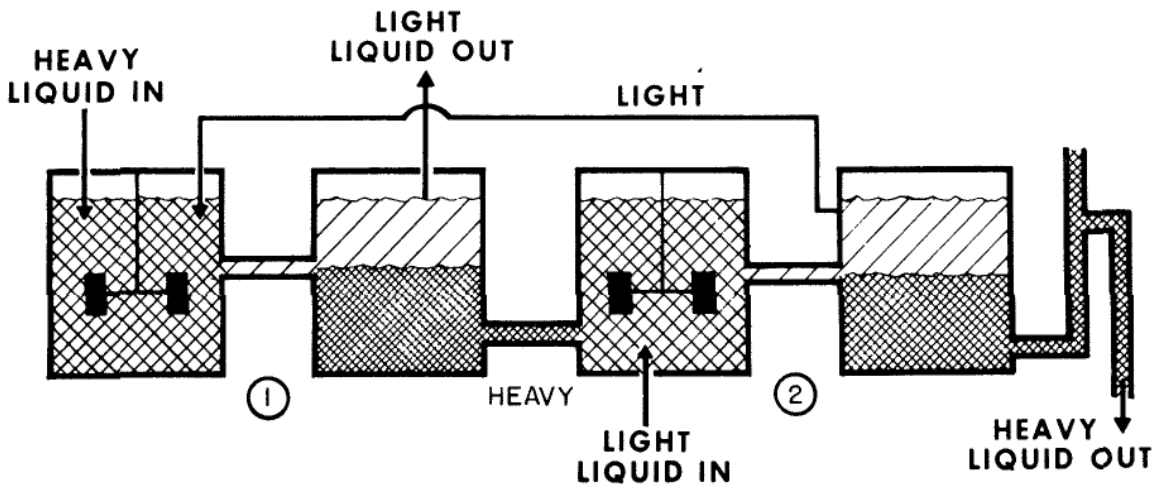

Fig. 5. Schematic of two-stage countercurrent mixer-settler.

emulsion to settle later on. It is usually a matter of providing sufficient flexibility in the equipment so that this maximum dispersion can be achieved and the desired performance realized.

Determining the capacity of a continuous multistage column almost always involves pilot scale testing, since small amounts of extraneous materials can affect the interfacial tension significantly. This affects the power of dispersion and maximum feed rate in the equipment. There are several methods available for estimating capacities of some types of equipment, but it must be remembered that these were obtained with relatively pure particles or liquids and industrial processing equipment will not always perform this way.

There is also a minimum size of pilot plant unit that can be used for predicting scale-up [14], since droplet size doesn't scale down in proportion and the point can be reached at which the physical size of the impeller blades and their associated fluid shear rates do not relate at all to the dispersion mechanism in the full-scale equipment—this can be compared to trying to hit a basketball with a baseball bat.

It is possible to run very small-scale equipment to obtain chemical extraction data, but this is quite different from using these data for mixer scale-up to large columns.

Mixer-settlers are used for extraction, and there is no reason that the approach to equilibrium in the mixer stage cannot equal one theoretical stage, given sufficient time and agitation level. The settler must be designed so that the emulsion produced from the mixing stage can be coalesced effectively.

There are applications for single-stage liquid-liquid contacting which may be done either at moderate power levels in large mixing vessels or with pipeline line blenders. The latter usually operate in short retention times but at higher power levels to give a very intimate two-phase dispersion.

Batch-type extractors are still quite common in many industrial processes, and there are a few new designs being prepared for these at the present time.

II. SINGLE-STAGE CONTACTING

A. Predicting the Dispersed Phase

Selker and Schleicher [15] give a relationship for predicting which phase will be the dispersed phase in a two-phase liquid-liquid system, depending upon the phase volume ratio and the viscosity ratio. For a given pair of liquids, there can be a wide range of relative phase volumes in which either phase could be the dispersed one. Which one will be dispersed depends upon the method of starting the system.

Referring to Fig. 6, two immiscible liquids are introduced batchwise into the tank and the impeller is placed in the center of one of the phases.

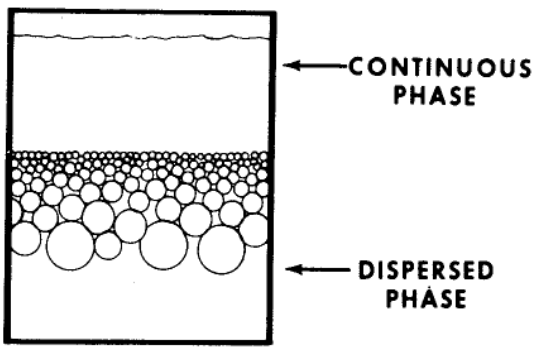

Fig. 6. Schematic illustration of phase separation from two-phase dispersion. The large drops are droplets of the dispersed phase collecting in the dispersed phase zone of the interface.

That phase will become the continuous phase and will remain so if that condition is a stable possibility. As the impeller is turned on from zero speed up to higher speeds, it will start to draw the other phase into that zone, and if the volume ratio and viscosity ratio allow, the dispersed phase will remain dispersed.

If one wants to place the impeller in the lower part of the tank and have

the heavy phase dispersed, the light phase must be added first, the impeller turned on, and then the heavy phase added to the system. This will normally keep the heavy phase dispersed if, again, it is a stable possibility.

Because results from mass transfer and reaction studies depend upon which phase is dispersed, it is very important to establish which is the dispersed phase and to start the tanks properly so the desired phase will be dispersed.

For further discussion on the prediction of the dispersed phase, see Quinn and Sigloh [16] and Rodger et al. [17].

B. Formation of Dispersions

Several investigators have measured the speed, diameter, and power of a mixer in producing a visually uniform dispersion over a variety of systems. In some cases, samples were used to determine the uniformity of the dispersion. There is so much variation in interfacial properties of industrial systems, due to impurities, that the use of pure liquid properties does not usually give satisfactory calculations.

Three investigators give equations for liquid-liquid systems in which water was one phase [1, 18, 19]. Non-Newtonian fluids were used by Gluz and Pavlushenko [20]. Two other investigators found that directing propeller flow by means of a draft tube was very effective [21, 22].

C. Drop Size

The distribution of droplet size in a liquid-liquid system under agitation involves both the production of droplets at the shear zone of the impeller and the possible collision coalescence of droplets in local shear portions of the tank.

On scale-up, the distribution of shear rates in large tanks is quite different from the distribution of shear rates in small tanks [23], and the correlation of effects is extremely complex.

Many experiments have been done with light transmission techniques, using transparent tanks and fluids. This, of course, does not work with most industrial systems, because fluids or vessels are usually relatively opaque. Most of the correlations use the physical geometry of the impeller, such as the diameter, speed, number of blades, and power level in the tank, and do not treat the fluid shear effects involved in the systesm. Therefore, the equations are only for the particular geometry tested and often only for the range of vessel sizes studied. The earliest work in this area was done by Rodger et al. [17] and Vermeulen et al. [24].

A survey of British industry gives an idea of the degree of concern that this type of problem creates [25].

Drop sizes in strongly coalesced liquids were measured by Sprow [26, 27]. Chen and Middleman [28] looked at drop size in very dilute systems, while Karam and Bellinger [29] set up theoretical equations and verified them experimentally.

Determination of droplet size using an encapsulation technique is a unique process [30]. Studies with rotating disks have reported actual experimental measurements [31, 32].

Relationships for drop size were given by Kagan and Kovalev [29, 33], while Pebalk and Mishev [34] looked at droplet size both with and without mass transfer.

D. Interfacial Area

A general review of the interfacial area for liquid-liquid processes was given by Pitt [35]. The use of chemical reactions to measure interfacial area was presented by Fernandez and Sharma [36]. A new method of measuring coalescence rates uses change in agitation intensity [37]. The use of controlled densities to separate, disperse, and coalesce bubbles has also been investigated [38].

Another study looked at the viscosity ratio of phases in a flow-type mixer [39]. Miller et al. [40] measured the rate of mixing within the dispersed phase and found that a typical mixing rate was 10 volumes of dispersed phase per minute at a power input of 10 hp/1000 gal.

E. Actual Plant Operations

Stemerding et al. [41], using radioactive tracer techniques on a 4000-gal vessel, traced the hydrocarbon and the aqueous streams simultaneously. They found that a propeller pumping upward resulted in improved operation over down-pumping. Other investigators determined the conditions under which the dispersed phase can form a separate layer, and its effect on this layer was studied [42].

F. Mass Transfer Studies

Schindler and Treybal [44] measured continuous phase mass transfer coefficients for ethyl acetate dispersed in water. Specific interfacial area was determined. It was found that the coefficients for unbaffled tanks were generally larger than for baffled tanks at the same horsepower.

Keey and Glen [45] investigated other organics in water and found that the dimensional relations derived from the postulate of locally isotropic turbulence may be used for translating mass transfer behavior.

Other investigators have correlated extraction rates in terms of Nusselt number-Reynolds number correlations [46] and a Prandtl number correlation [47].

Rushton et al. [48] used a sudden addition of a solute to a batch dispersion and measured the time change of solute concentration in the continuous phase to determine mass transfer coefficients in different tank sizes. Surface renewal relations have been studied with separately stirred phases [49-51].

Other studies have investigated overall mass transfer rates [52] and mass transfer rates in a contactor in which interfacial area could be measured [53].

The effect of surface-active agents was measured and the experimental data were correlated to a relationship describing the appearance of a current of dye and the distribution coefficient for the dye [54]. Kremnev et al. [55] used a relationship involving the surface tension before and after adding the surface-active compound.

Studies involving chemical reactions include surveys by Rietema [56] and Oshima and Miyauchi [57]. A theoretical study by Curl included the effect of mixing on second-order by-product reactions [47].

Among the chemical systems studied are oxidation reduction rates with tetravalent cerium and tetrachlorohydroquinone [58], ethyl acetate ethyl formate and caustic [59], toluene and sulfuric acid-nitric acid mixtures [19], and emulsion polymerization of epoxies [60].

Abramson et al. [61, 62] used separate stirrers in two phases to transfer iodine from organic solutions into aqueous thiosulfate and orthonitrophenol into aqueous caustic; these workers gave detailed data on reaction mechanics.

Other investigators have looked at extraction with and without reaction [63] and have used high speed dispersion homogenizers [64]. Shain [65] and Spielman and Levenspiel [66] used various mathematical models to describe the dispersion, coalescence, and reaction in two-phase systems.

A single-stage paddle mixer was used in studies which correlated the mass transfer coefficient in terms of continuous phase Prandtl number [67-69].

III. CONTINUOUS COUNTERCURRENT EXTRACTORS

A. Holdup

Two studies gave equations for the holdup in a variety of agitated columns. A graph is presented for a rotating disk contactor [70], and equations are given by Misek for a variety of column designs [71]. Experimental results in a rotating disk contactor verified the concept that drops of dispersed liquid in an agitated system move vertically on the average, with a velocity equal to the terminal velocity of rigid spheres [72]. Misek [73] reported results that can be used for estimating the terminal drop size in rotating disk contactors and other agitated devices.

B. Axial Mixing

There are two methods of looking at axial mixing in continuous extractors. The stage model using a series of "completely mixed" tanks connected by interstage mixing is illustrated in Fig. 7. Figure 8 shows the effect of interstage mixing on concentration driving force for five theoretical stages. The residence time of the equipment needed to obtain five

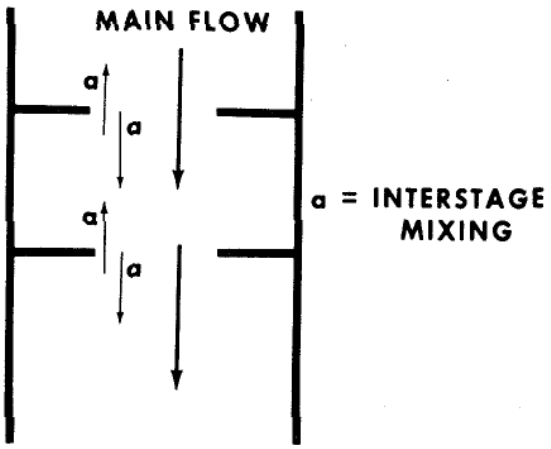

Fig. 7. Schematic illustrating the concept of the volume of a fluid having interstage mixing in addition to the main flow through the stage.

theoretical stages would have to be longer in the case of interstage mixing compared to the case of no interstage mixing. Several authors use the concept of an axial mixing or diffusion coefficient, which is correlated by means of a Peclet number. Relations are available to convert from one model to another [74].

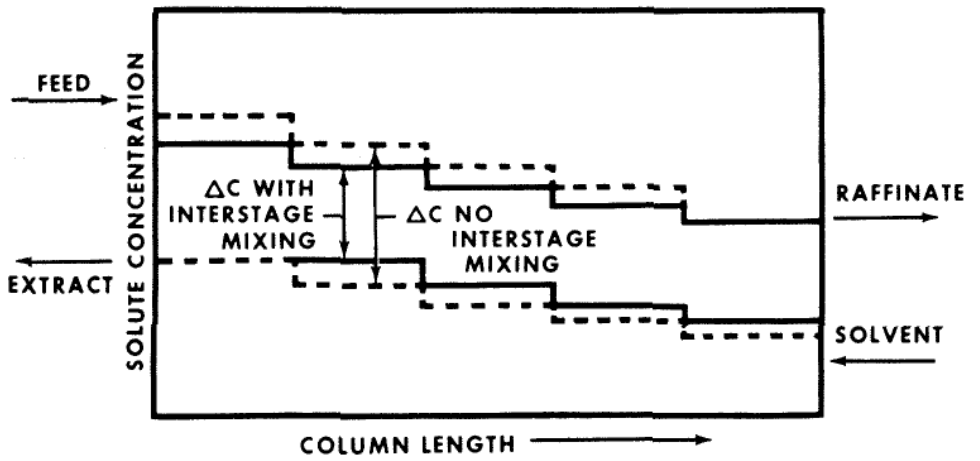

Fig. 8. Schematic illustrating the effect of interstage mixing in reducing the the effective Δc driving force.

Bibaud and Treybal [75] used this axial diffusion concept with an Oldshue-Rushton column even though it has separate stages. They gave an equation* for the continuous phase back-mixing coefficient, E_C:

$$\frac{(1 - H)E_C}{V_C Z} = 0.268 \left(\frac{DN\,(1 - H)}{V_C} \right) - 0.14 \qquad (1)$$

Another equation was developed for the dispersed phase, E_D:

$$\frac{D^2 N}{E_D} = 3.93 \times 10^{-8} \left(\frac{D^3 N^2 \rho_C}{\sigma} \right)^{1.54} \left(\frac{\rho_C}{\Delta\rho} \right)^{4.18} \left(\frac{D^2 N \rho_C}{\mu_C} \right)^{0.61} \qquad (2)$$

Gutoff gives relations for the continuous phase interstage mixing in an Oldshue-Rushton column [76]. Goncharenko and Gotlinskaya [77] determined residence time data for continuous and dispersed phases in a Scheibel column.

Rod [78] gave an equation for the diffusional component of longitudinal mixing in the dispersed phase of a rotating disk column.

Stermerding et al. [79] present

*A list of the symbols used in the following is given in Sec. IX of this paper.

$$E_C = 0.5ZV_C + 0.12\,DNZ\left(\frac{D_o}{T}\right)^2 \qquad (3)$$

as the relation for continuous phase axial mixing in a rotating disk column.

A general survey of axial mixing is given by Vermeulen et al. [80]. With two different diameters of rotating disk contactors it was found that the coefficient of axial mixing is approximately the same as the coefficient of linear mixing of the continuous phase [81].

Gel'perin et al. [82] described longitudinal diffusion as equal to the sum of the back-mixing and axial diffusion and studied this phenomenon in a rotating disk contactor, a Scheibel extractor, and a mixer-settler.

Back-mixing was studied by Stainthorp and Sudall [83]. Kagan found that the longitudinal coefficient of the mixing increases with the diameter of the apparatus [84].

Studies of the dynamic response of extraction columns include a review [85] and a study that describes an apparatus for carrying out these experiments [86].

C. Performance Data

There are data on many types of systems for almost all of the presently used contactors. The following is a breakdown of the available data according to column type.

1. Rotating Disk Contactor. One literature review gives plots between the number of theoretical plates and various parameters [87]. Another review describes the use of the rotating disk contactor in a laboratory program [88]. Other authors have described an extraction in which the physical properties change appreciably during extraction and the dimensions of each section should be adjusted accordingly [89]. Aerov et al. [90] looked at pilot plant testing and the change in HTU* with column diameter. Misek [91] showed that when the separation coefficient is less than 1 it is preferable that the feed be dispersed.

Ponikarov et al. [92] defined an effectiveness factor as the throughput divided by the HETS, and gave values for a variety of rotating disk column geometries. The use of perforated rotors in a rotating disk contactor seemed to improve mass transfer rates by giving reduced throughput [93].

2. Scheibel Column. One study on the Scheibel column used a system

*See Sec. IX for definition of HTU and HETS.

of propionic acid, water, and toluene and reported relationships between impeller speed and throughput [94].

3. Oldshue-Rushton Column. Multistage units of the Oldshue-Rushton type have also been studied [95-97].

A very extensive investigation by Gel'perin et al. [98] compared data from a modified Scheibel column and a modified Oldshue-Rushton column with earlier existing data for a rotating disk contactor. Several plots and correlations were given.

D. Stage Construction

With Oldshue-Rushton columns, the stage efficiency tends to increase with scale-up, since a greater liquid depth is used in the larger columns (Fig. 9.).

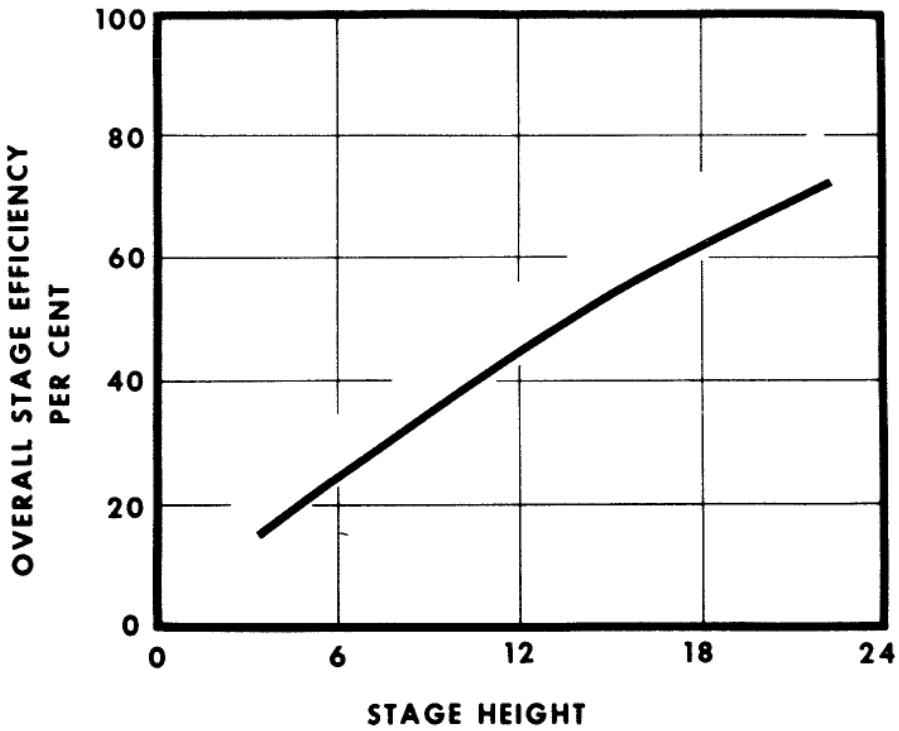

Fig. 9. Effect of stage height on stage efficiencies for Oldshue-Rushton column.

The effect of flow turndown is very important. In systems with higher $K_L a$, decreasing the throughput lowers column performance. This is due to the increased relative interstage mixing, and the longer residence time is not helpful (Fig. 10).

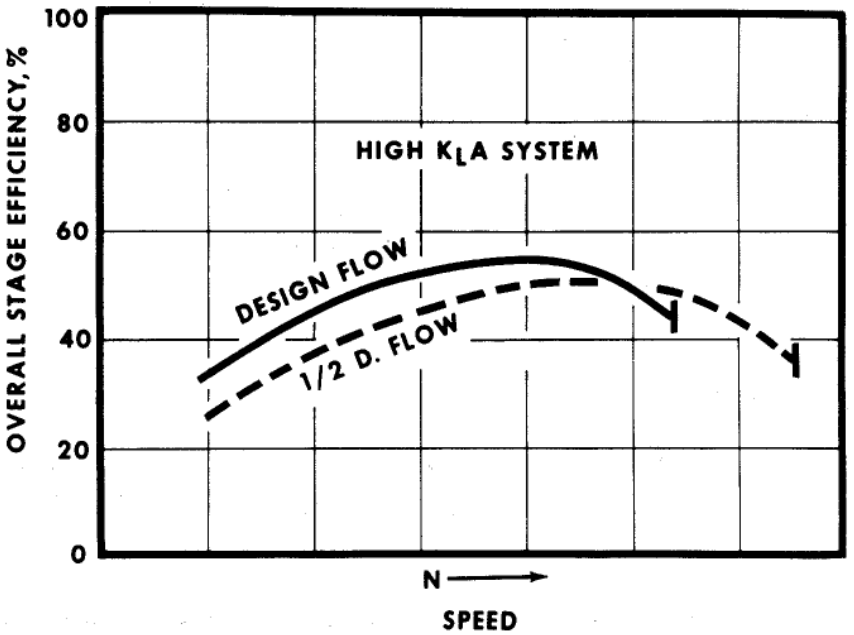

Fig. 10. Effect of throughput turndown ratio on stage efficiency for high K_La system.

In low K_La systems, the increased residence time is advantageous and outweighs the adverse effects of increased interstage mixing; the result in Fig. 11 is obtained.

Treybal [97] described a column with special calming zones between mixing stages to achieve an unusually low value of HETS.

Modifications for improving the performance of a rotating disk contactor are described in Ref. [99]. The Morris contactor is used for hard-to-settle dispersions [100].

An impeller with alternate oppositely pitched blades was used to set up wave pulses [101]. Other investigators studied the pulsing nature of impeller flow in an extraction column [102]. The addition of rotating impellers to a pulsed sieve plate column gave several advantages [103]. The Humboldt-Ziehl column has both rotation and vertical movement [104, 105].

IV. MIXER-SETTLERS

A good summary of methods used for mixer-settlers was given by Treybal [106]. He presents some of the techniques used to obtain mass

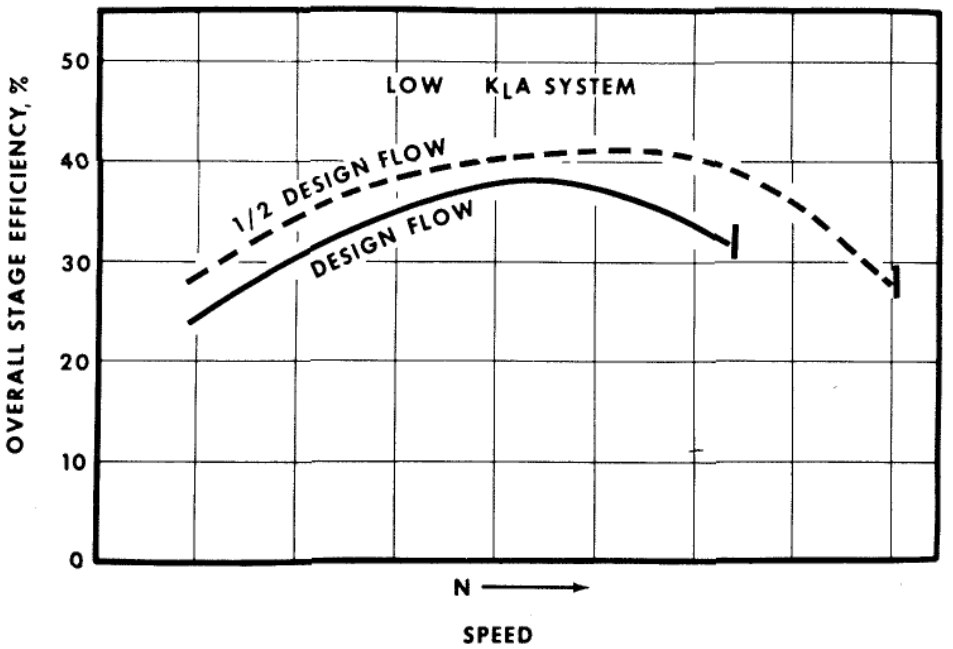

Fig. 11. Effect of turndown ratio on stage efficiency for low K_La system.

transfer coefficients in mixer tanks. In general, there is no reason that practically 100% stage efficiency can't be obtained with sufficient residence time and mixer power in the mixer, and sufficient residence time in the settler. Another article by Treybal treats the recycle characteristics in extraction [107]. Figure 8 shows a typical two-stage system.

Coughlin and Von Berg determined mass and heat transfer coefficients for kerosine and dispersed water for 2-ethylbutric acid, a solute [108]. Design principles for large mixer-settlers have also been presented [109].

Berestovoi and Romankov derived several dimensionless equations with nine different systems using three different liquids [110].

The mechanical operation of a mixer-settler was described in Ref. [111].

It is often an advantage to use the mixer as a pump, to pump one phase through a mixer-settler apparatus as well as mixing that phase in the mixing zone. The other phase can be arranged to flow by gravity [112, 113]. Performance data on "box extractors" are used to get a large number of theoretical stages, and several reports are now available on the operation of these units [114-116].

The design of mixer-settler extractors for the particular case of rare earth elements illustrates the use of mathematical and experimental planning [117].

Horizontal mixers are used in centrifuge mixer-settler combinations, and

performance data on several systems are now available [118, 119]. Holdup [120, 121], the height of a dispersion band in a settler [122], and analysis of coalescence [123] are all related to mixer-settler performance.

Other investigators have looked at specific processes including the removal of phenol from tar water [124]; nuclear fuels [125]; and the use of ammonia as a solvent [126].

V. MISCELLANEOUS EXTRACTORS

A. Horizontal Shafts

Several extractors use a horizontal shaft, which usually involves mixing and calming sections. Publications dealing with this type of unit include work on a variety of liquid systems [127-130]. One such unit used a rotating nozzle [131]. Gel'perin et al. [129] looked at both mass transfer and longitudinal mixing in units with horizontal shafts.

B. Laboratory Units

Extractors designed for laboratory use include a rotating disk column [132, 133], a box-type settler [134], a mixer-settler [139], a single-stage unit [135], and a mixer-centrifuge combination [136]. Two analytical techniques have been presented, one using a mixer-centrifuge and the other using a device for analytical detection [137, 138].

C. Fluidized Droplet Reactor

A detailed description [133] has been given of scaling up a fluidized droplet reactor, using 8-in.-diameter and 40-in.-diameter reactors, to study catalyst concentration and mass transfer effects. Residence time and linear fluidized droplet velocities make it difficult to scale up to large size reactors.

VI. STUDIES ON SPECIFIC CHEMICAL SYSTEMS

A. Alkylation

Mayer [140], using four horizontal, geometrically similar tanks that ranged from 8 in. to 6 ft in diameter, found a correlation for estimating the mixing time required for forming emulsions. Sprow investigated the role of interfacial area in determining alkylate quality [141]. Fedorvskii

et al. [142] analyzed samples of reaction mixture from the reaction zone to control the alkylation process.

Shlegeris and Albright [143] studied both acid- and oil-base emulsions in the alkylation process. The use of settling behavior as a guide to emulsion quality was described by Jernigan et al. [144].

By the use of radiotracers, Hull et al. measured circulation rate, volume of acid, replacement rate of acid, and entrainment of acid hydrocarbon stream [145].

The effect of operating variables with high agitation rates and short contact times was studied by Mosby and Albright [146].

B. Other Processes

Performance data obtained from a variety of equipment have been given for a number of processes including dephenolization of waste water [147-149], production of caprolactam [150, 151], acid naphthalene mixtures [152], acid washing [153], aromatic hydrocarbons [154], the Amex solvent extraction process [155], and recovery of vanadium from a waste stream [156].

VII. REVIEWS AND PATENTS

Many reviews appear each year; Treybal [157] gives a good summary of publications up to 1963. Symposia held in 1966 reported on equipment in the United States [158] and abroad [159], and presented recent technology of columns and mixer-settlers [106].

The most recent reviews are those by Hanson [160-163], Schreiner [164], Mumford [165], and Jeffreys [166]. Other reviews from previous years are listed in the References [3, 4, 83, 87-92, 107, 111, 127, 132, 148, 167-178]. About 50 recent patents on liquid-liquid extraction mixing equipment are also listed [5-12, 179-227].

VIII. CURRENT PRACTICE

Data on sales and installations of various types of extraction equipment are not published.

It appears that in the mining industry there are many mixer-settlers being installed. Usually land area is not at a premium and the flexibility gained

by having a large number of separate tanks and mixers is very desirable for these types of processes. A disadvantage is the additional maintenance required on many separate items of equipment. The major advantage is the ability to put in one or two extra tanks to provide for periodic shutdown and maintenance of individual equipment items.

There are several advantages to using the pumping action of a mixer, rather than pumps, to pump fluids from tank to tank in a mixer-settler system. Pump-mixer settlers are also currently being designed and installed.

In the petroleum industry, the vertical multistage rotating disk contactor unit has been widely accepted for a variety of processes.

The York-Scheibel column and the Oldshue-Rushton column are more commonly used in the chemical and mining industry. There is a tendency to use vertical columns with mechanical mixers in contrast to stationary internal baffles when going to new extraction processes. However, there are still many units of the traditional baffle or tray type being used and installed in existing processes.

The governing factor in larger vertical multistage equipment is the cost involved in manufacture and maintenance, and there has not been wide acceptance as yet of newer proposed columns which add various types of asymmetric impeller positioning or internal baffle and coalescing sections.

On many applications, both mixer-settlers and vertical multistage columns are designed and estimated, and a project evaluation is made to determine which system is best for the particular installation at hand.

IX. NOMENCLATURE

D	impeller diameter
D_O	diameter of opening between stages
E_C	back-mixing coefficient, continuous phase
E_D	back-mixing coefficient, dispersed phase
H	holdup, fractional dispersed phase
HETS	height equivalent to a theoretical stage
HTU	height of a transfer unit
K_La	mass transfer coefficient, (mass transfer rate)/(concentration driving force)
N	rotational speed
T	tank diameter
V_C	mean superficial velocity, continuous phase
V_D	mean superficial velocity, dispersed phase

Z	height of a compartment
$\Delta\rho$	difference in density between continuous and dispersed phase
σ	interfacial tension
ρ_c	density, continuous phase
μ_c	viscosity, continuous phase

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