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STRUCTURED PACKINGS IN LIQUID-LIQUID EXTRACTION

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ABSTRACT

Extractors equipped with structured packing are becoming more important in the chemical process industries. These devices provide high mass transfer efficiency and capacity relative to random packings and sieve trays. At the present time, many sieve tray extractors are being retrofitted with structured packings to enhance mass transfer efficiency and capacity. This paper will present a comparison of the performance of structured packing with sieve trays, some background on the commercial development of structured packings, and fundamental models required to design a liquid/liquid extractor equipped with structured packing.

INTRODUCTION

Extraction is typically carried out using a variety of devices to enhance contacting of the two liquid phases. In liquid-liquid extraction, there are various nonproprietary types of devices including sieve trays and random packing and structured packings. The sieve-tray column is a widely used, nonproprietary extractor. The sieve holes allow drop reformation and the continuous phase is moved through downcomers (or upcomers). One can imagine the sieve-tray extractor as a series of short spray columns where backmixing of the continuous phase is constrained within the sieve-tray compartment.

In the packed column, the packing elements provide a tortuous path for the dispersed-phase drops and promote drop breakage, thus increasing the interfacial area for mass transfer. The packing elements also reduce backmixing of the continuous phase. As shown Figure 1, the packed column consists of multiple packed beds with intermediate redistributors. Two types of packings are used in liquid/liquid extraction—random and structured packings. In recent years, structured packings have become more popular, replacing both sieve trays and random packing. The ordered nature of the structured packing is thought to reduce channeling and phase maldistribution which is common in large-scale columns, particularly with random packing. The use of structured packings in extraction developed from distillation applications. These packings are now being used to achieve the same improvements in efficiency and capacity in liquid-liquid extraction.

PREVIOUS WORK

Relative to a sieve-tray column, a structured sheet metal packing can provide a 30% improvement in efficiency without reducing capacity (1,2). This comparison is shown in Figure 2. Examples of extraction columns which have now been retrofitted with structured packings are given in Table 1 (3). In this paper, the parameters involved in the design of extractors with structured packings are discussed and methods of their calculation are presented.

DESIGN WITH STRUCTURED PACKINGS

The height of a differential contactor (Z) may be calculated using the NTU and HTU method as shown in Equation 1. The number of transfer units is calculated from the phase equilibria and the separation. Methods of NTU calculation are presented by Treybal (4). Equation 2 may be used for the case of a dilute solute composition and a straight equilibrium line.

$$Z = NTU_{oc} \bullet HTU_{oc} \tag{1}$$

$$NTU_{oc} = \frac{\ln\left\{\left(\frac{x_f - y_s/m}{x_r - y_s/m}\right)\left(1 - \frac{1}{\lambda}\right) + \frac{1}{\lambda}\right\}}{\left(1 - \frac{1}{\lambda}\right)} \tag{2}$$

The height equivalent to a theoretical stage (HETS) is often used to compare the efficiency of staged and differential contactors. This term is used by vendors

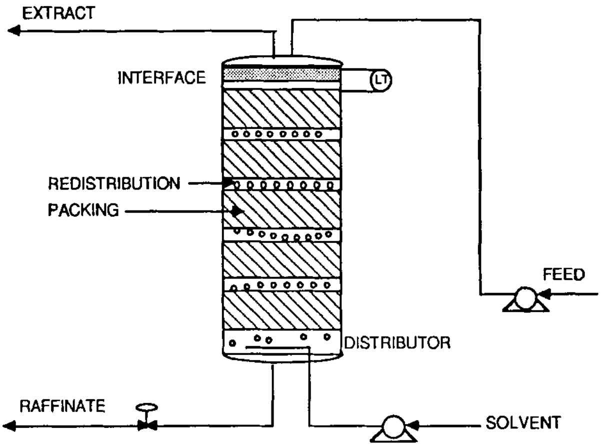

FIGURE 1. Packed Extractor

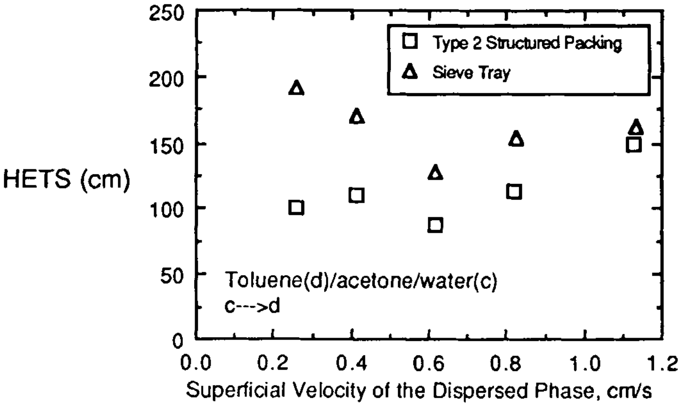

FIGURE 2. Comparison of Sieve Trays with a Type 2 Structured Packing. System: toluene(d)/acetone/water(c). S/F=1.05. Direction of Mass Transfer: c--->d. Column Diameter=0.425 m.

Table 1. Industrial Liquid Extractors Equipped with Structured Packings (3)

Applications	Column Diameter [mm]	Packing Height [m]
Extraction of Nitrotoluene after reaction of HNO3 with Toluene in H2SO4	600	12
Extraction of Methylacrylate from organic solution with Perchlorethylene	150	9
VI-reaction column: Cyclohexanone/Ammoniumhydrogen-Sulfate-solution	1100	18
Extraction of Furan from an aqueous solution with Perchlorethylene	300	10
Benzylalcohol from a salt solution with Toluene	500	12
Extraction of Sulfolane from C6C7-mix with water	420	9
Removing H2S from LPG with MDEA	2350	12
Extraction of Caprolactam from Ammoniumsulfate-solution with Benzene	990	10
Removing Ammoniumsulfate from Benzene/Caprolactam-solution with water	1450	10
Extraction of Imidazol from an aqueous solution with an organic solvent	650	14 + 8
Extraction of Acrylic acid from waste water with Butanol	800	10 + 6
Washing column Oil/Water	700	6
Extraction of Propylenechlorhydrin from Dichloropropane with water	1000	22
Removing H2S from LPG with MDEA	1200	10
Removing H2S from LPG with DEA	870	4
Extraction of C-4/C-4 HCl from C5/Heptane with aqueous hydrochloric acid	470	7
Extraction with water	150	6.1
Removing H2S from LPG with MDEA	1100	8.5
Extraction of flavour agent from an aqueous solution with Alkane	250	5
Removing residual Alkali from Dichlorohydrazobenzene with water	450	4.5
Extraction of Methanol from LPG with water	730	11.4
Extraction of Methanol from LPG with water	1470	7.8
Removing H2S from LPG with MDEA	1700	3.2
Extraction of Chloroacetic acid from Methylchloroacetate with water	300	3

because the contacting height is easily calculated when the number of theoretical stages is known. It is important to note that the height of a transfer unit (HTU) and the HETS are not the same except when the extraction factor is unity. The relationship between the two is given in Equations 3 and 4

$$\mathrm{HETS} = \frac{\lambda \ln \lambda}{\lambda - 1}\,\mathrm{HTU}_{oc} \qquad \text{for } \lambda \neq 1 \tag{3}$$

$$\mathrm{HETS} = \mathrm{HTU}_{oc} \qquad \text{for } \lambda = 1 \tag{4}$$

where λ is the extraction factor defined in Equations 5 and 6.

$$\lambda = m_{dc}\left(U_d / U_c\right) \tag{5}$$

$$m_{dc} = \frac{C_d^*}{C_c} \tag{6}$$

Hydrodynamics (Capacity)

An understanding of the hydrodynamics of a contactor is critical for the design of the diameter and height of the extraction column. The hydrodynamics affects the capacity and mass transfer efficiency of the internals within the extractor. An accurate prediction of drop diameter, dispersed-phase holdup, slip velocity, and flooding velocities are necessary for the optimum design of the column height and diameter.

In order to design an extractor, one must first determine the average drop diameter of the system. For the case of the packed extractor, models have been developed based primarily on photographic studies of droplet dispersion. We have observed that the Sauter mean drop diameter in the packed extractor is dependent on the system properties such as interfacial tension and density difference, and is essentially independent of the type and geometry of the packing (2). The Sauter mean drop diameter may be calculated from Equation 7. The drop diameter was also observed to be dependent on the direction of mass transfer (2). In general as a result of Marangoni effects, larger drops are formed when solute is transferred from the dispersed to the continuous phase due to enhanced coalescence.

$$d_{vs} = 1.15\,\eta\sqrt{\frac{\sigma}{\Delta\rho\, g}} \tag{7}$$

$$\eta = 1.0 \quad c \rightarrow d$$

$$\eta = 1.4 \quad d \rightarrow c$$

Once the mean drop size has been determined, the average velocity of the drops may be calculated. The capacity of any extraction device is dependent on the speed at which the dispersed-phase drops travel through the column. The average velocity of the dispersed-phase drop is much greater than the average velocity of the dispersed phase through the empty column, and it can be calculated from Equation 8.

$$U_{drop} = \frac{U_d}{\varepsilon\phi_d} \tag{8}$$

Likewise, the interstitial velocity of the continuous phase is greater than the average velocity of the continuous phase through an empty column. The relative velocity between the phases is known as the slip velocity U_S and may be calculated from Equation 9.

$$U_S = U_{drop} + U_{ic} \tag{9}$$

The speed of the dispersed-phase drop, relative to the continuous phase, can be determined from a fundamental force balance on a drop with a mean diameter of d_{VS}. Figure 3 gives a force balance on a drop. Gravitational, buoyancy, and drag forces are considered.

$$F_{buoyancy} - F_{gravity} - F_{drag} = 0 \tag{10}$$

where

$$F_{buoyancy} = \rho_c\left(\frac{\pi}{6}\, d_{vs}^3\right) g \tag{11}$$

$$F_{gravity} = \rho_d\left(\frac{\pi}{6}\, d_{vs}^3\right) g \tag{12}$$

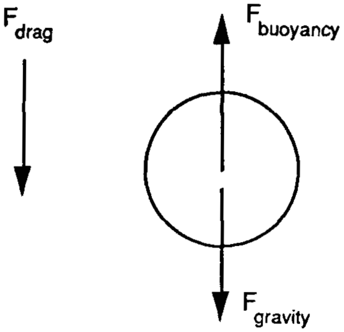

FIGURE 3. Force Balance on a Drop

$$F_{drag} = \frac{1}{2} C_D \rho_c \left(\frac{\pi}{4} d_{vs}^2 \right) U_{so}^2 \tag{13}$$

Re-arranging Equations 9 to 12 allows the determination of the characteristic slip velocity U_{SO}, given in Equation 14. The characteristic slip velocity must be corrected for the effect of the dispersed-phase holdup and the nature of the contacting device to give the actual slip velocity between the phases. The generalized slip velocity U_S model is given in Equation 15 (2).

$$U_{so} = \left[\frac{4 \Delta\rho\, g\, d_{vs}}{3 \rho_c C_D} \right]^{0.5} \tag{14}$$

$$U_s = U_{so} \exp(-1.92\, \phi_d) \cos\left\{\frac{\pi\zeta}{4}\right\} + \left(1 - \cos\left\{\frac{\pi\zeta}{4}\right\}\right)\left(\frac{U_c}{1 - \phi_d}\right) \tag{15}$$

where the tortuosity factor ζ is defined as

$$\zeta = a_p \bullet d_{vs} / 2 \tag{16}$$

Because of the drag coefficient term, the calculation of U_{SO} by Equation 14 can be tedious. The model of Grace, Waireyi, and Nguyen (5) may be used to calculate the characteristic velocity. The required equations are given below.

$$\frac{N_{Re}}{P^{0.149}} = 0.94\,H^{0.757} - 0.857 \qquad H \le 59.3 \tag{17}$$

where

$$\frac{N_{Re}}{P^{0.149}} = 3.42\,H^{0.441} - 0.857 \qquad H > 59.3 \tag{18}$$

$$P = \frac{\rho_c^2\,\sigma^3}{\mu_c^4\,g\,\Delta\rho} \tag{19}$$

$$H = \left[\frac{4\,d_{vs}^2\,g\,\Delta\rho}{3\sigma}\right]\left[\frac{\mu_w}{\mu_c}\right]^{0.14} P^{0.149} \tag{20}$$

$$\mu_w = 0.9\ \mathrm{cp}$$

$$N_{Re} = \frac{d_{vs}\,\rho_c\,U_{so}}{\mu_c} \tag{21}$$

The fraction of the void volume occupied by the dispersed phase in the extractor is an important design parameter. Dispersed-phase holdup can be calculated implicitly from Equation 22 (2).

$$\phi_d = \frac{U_d \cos^{-2}\left(\frac{\pi\zeta}{4}\right)}{\varepsilon\,U_{so}\exp\left(-1.92\phi_d\right) - \frac{U_c}{1-\phi_d}} \tag{22}$$

In packed extractors, capacity or flooding occurs when the phases are no longer moving in a countercurrent fashion through the column or when the column is no longer operational. For example, drops may be entrained with the continuous phase, or a light dispersed phase may continue to accumulate beneath a packing support and eventually exit the bottom of the column. Another type of flooding condition that usually occurs at high dispersed-to-continuous-phase ratios is phase inversion. During phase inversion, the dispersed droplets pack tightly together and coalesce. The former dispersed phase becomes the continuous phase. The interface control is then located on the wrong end of the tower, thereby disabling the operation of the extractor.

We developed a fundamentally consistent flooding model for packing utilizing the dispersed-phase holdup equation. By assuming that during flooding drops are packed together in a cubic arrangement as shown in Figure 4, a maximum holdup of $\pi/6$ is possible. By substituting $\pi/6$ or 0.52 in the ϕ_d terms of the holdup equation, the following general flooding model was developed.

Flooding in packed extraction columns can be predicted using the following generalized equation. This equation is derived strictly from physics and is not developed by empirical correlation of data.

$$\frac{1}{U_{cf}} = \frac{5.63}{\varepsilon U_{so}} + 5.21 \left[\cos\left\{ \frac{\pi \zeta}{4} \right\} \right]^{-2} \left[\frac{\left(U_{df} / U_{cf} \right)}{\varepsilon U_{so}} \right] \tag{23}$$

Another advantage of this model, which is a result of its mechanistic development, is if one applies it to the limit to low packing areas, Equation 24 may be obtained. Empirically derived flooding models for packed columns reported by others usually predict infinite flooding rates if this condition is applied. This is very unfortunate since many of the empirical models are derived by regression of data bases which are heavily weighted with flooding data obtained from small columns which use high-surface-area packings. Therefore the advantage of this approach is that the presented flooding model will be applicable to extrapolation to lower-surface-area packings commonly used in industry.

$$\text{limit } a_p \to 0 \qquad \frac{1}{U_{cf}} = \frac{5.63}{U_{so}} + 5.21 \left[\frac{\left(U_{df} / U_{cf} \right)}{U_{so}} \right] \tag{24}$$

Where U_{cf} and U_{df} are the superficial velocities for the continuous and dispersed

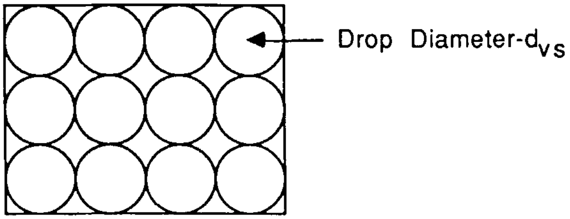

FIGURE 4. Maximum Dispersed-Phase Holdup

phases at the flooding point. The column diameter may be calculated from the flooding velocity and the design fraction of flood, β_f.

$$\text{Column Diameter} = \sqrt{\frac{4Q_c}{\pi \beta_f U_{cf}}} \tag{25}$$

The mass transfer efficiency of most extraction devices is proportional to the area available for mass transfer. For the case where the dispersed phase travels through the column in the form of drops, the interfacial area can be calculated from Equation 26.

$$a = \frac{6 \varepsilon \phi_d}{d_{vs}} \tag{26}$$

Mass Transfer

In liquid extraction, unimolecular, unidirectional diffusion is assumed to be the mechanism of mass transfer. The solute is assumed to be the only molecular species transferred between the immiscible phases. In order to accomplish this transference, the solute must overcome certain resistances: 1) movement from the bulk of the raffinate phase to the interface, 2) crossing the interface, and 3) movement from the interface to the bulk of the extract phase. If motion in the two phases dies out near the interface and the entire resistance to transfer is considered to be contained in two fictitious films on each side of the interface whereby mass transfer occurs by diffusion, then two-film theory can be applied. It is also assumed that local equilibrium exists at the interface and that the concentration gradients are established so rapidly in the films that steady-state diffusion can be assumed.

The rate of mass transfer at steady state (N) can be expressed in different forms as shown in Equations 27 and 28

$$N = K_{od}\,A\,(C_d^* - C_d) = K_{oc}\,A\,(C_c - C_c^*) \tag{27}$$

$$N = k_d\,A\,(C_{d,i} - C_d) = k_c\,A\,(C_c - C_{c,i}) \tag{28}$$

where A is the interfacial area, C_i indicates the concentration at the interface, and C^* indicates the equilibrium concentration. The overall mass transfer coefficient based on the continuous phase can be calculated from the individual resistances as given in Equation 29.

$$\frac{1}{K_{oc}} = \underbrace{\frac{1}{k_c}}_{\substack{\text{Continuous}\\\text{Phase}\\\text{Resistance}}} + \underbrace{\frac{1}{m_{dc}\,k_d}}_{\substack{\text{Dispersed}\\\text{Phase}\\\text{Resistance}}} \tag{29}$$

Likewise, the overall mass transfer coefficient based on the dispersed phase can be calculated from Equation 30.

$$\frac{1}{K_{od}} = \underbrace{\frac{1}{k_d}}_{\substack{\text{Dispersed}\\\text{Phase}\\\text{Resistance}}} + \underbrace{\frac{m_{dc}}{k_c}}_{\substack{\text{Continuous}\\\text{Phase}\\\text{Resistance}}} \tag{30}$$

In Equations 29 and 30, m_{dc} is the slope of the equilibrium line with the equilibrium concentration in the dispersed phase plotted on the ordinate and the concentration of the solute in the continuous phase plotted on the abscissa. The individual mass transfer coefficients for the dispersed and continuous phases are k_d and k_c, respectively.

For molecular diffusion, k_d and k_c are functions of the diffusion coefficients and thicknesses of the stagnant films near the interface, but in reality these coefficients are corrected for convection and turbulence effects. Two-film theory predicts that the mass transfer coefficient is directly proportional to the diffusion coefficient to the power unity. In contrast to two-film theory, other theories such as surface renewal show the effect of the diffusion coefficient to be to the 0.5 power. Experimental data generally appear to favor surface renewal theory.

In any extraction process, the mass transfer process into or out of a drop can happen in three stages: drop formation (N_f), drop rise (N_r), and drop coalescence (N_c). Total mass transfer is taken as:

$$N_t = N_f + N_r + N_c \tag{31}$$

Mass transfer during drop coalescence is generally negligible. With the exception of the case of the sieve-tray extractor, mass transfer during drop formation is also negligible. The majority of the mass transfer occurs during drop rise. The recommended individual continuous-phase mass transfer coefficient model (k_c) for drop rise is given below.

For the continuous phase,

Continuous-Phase Mass Transfer Coefficient Model (2)

$$k_c = 0.698\left(\frac{D_c}{d_{vs}}\right)Re_c^{0.5}\,Sc_c^{0.4}\left(1 - \phi_d\right) \tag{32}$$

where

$$Re_c = \frac{\rho_c U_s d_{vs}}{\mu_c} \tag{33}$$

$$Sc_c = \frac{\mu_c}{\rho_c D_c} \tag{34}$$

The theoretical model given above is very similar to the empirical model developed by Treybal in the correlation of the Ruby and Elgin (6) spray extraction data.

$$k_c = 0.725 \left(\frac{D_c}{d_{vs}}\right) Re_c^{0.57}\, Sc_c^{0.42} \left(1 - \phi_d\right) \tag{35}$$

The recommended equations for the dispersed-phase film mass transfer coefficient are given below. The condition for the proper selection is given in Equation 37.

For the dispersed phase

Dispersed-Phase Mass Transfer Coefficient Model

$$k_d = 0.023\, U_s \left[\frac{\rho_d D_d}{\mu_d}\right]^{0.5} \qquad \Phi > 6 \tag{36}$$

(Laddha and Degaleesan (7))

$$k_d = \frac{0.00375\, U_s}{1 - \mu_d/\mu_c} \qquad \Phi < 6 \tag{37}$$

(Handlos and Baron (8))

where

$$\Phi = \left(\frac{\mu_d}{\rho_d D_d}\right)^{0.5} \Big/ \left(1 + \mu_d/\mu_c\right) \tag{38}$$

In summary, the design of a packed extractor would involve the following steps for the dilute solute case.

(1) Obtain physical properties: densities, viscosities, diffusion coefficient, interfacial tension, equilibrium distribution coefficient, solvent and feed rate

(2) Obtain packing characteristics: surface area, void fraction

(3) Determine phase to be dispersed

(4) Calculate number of transfer units based on feed and separation requirements (Equation 2,5,6)

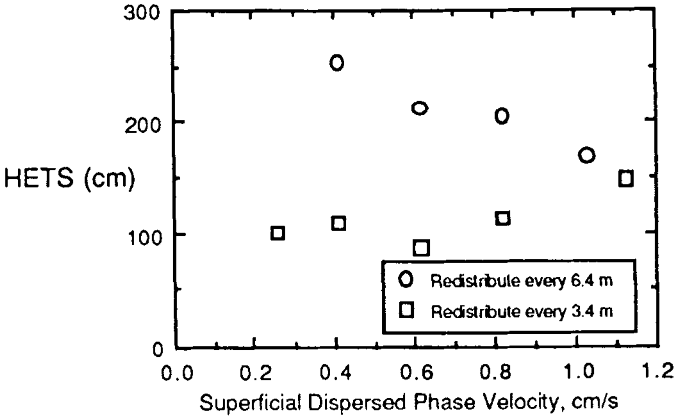

Figure 5. Effect of Packed Bed Height. System: toluene(d)/acetone/water(c). S/F=1.05. Direction of Mass Transfer: c→d. Packing: Type 2 Structured Packing. Column Diameter=0.425 m.

(5) Calculate Sauter mean drop diameter (Equation 7)

(6) Calculate characteristic slip velocity (Equations 17-21)

(7) Calculate flooding velocity (Equations 16, 23)

(8) Calculate column diameter based on design % of flood (Equation 25)

(9) Calculate superficial velocity of each phase based on flowrate and column diameter

(10) Calculate dispersed-phase hold-up (Equation 16,22)

(11) Calculate slip velocity (Equation 15)

(12) Calculate continuous-phase film coefficient (Equation 32-34)

(13) Calculate dispersed-phase film coefficient (Equation 36-38)

(14) Calculate overall mass transfer coefficient (Equation 29)

(15) Calculate interfacial area for mass transfer (Equation 26)

(16) Calculate volumetric mass transfer coefficient, $K_{oc}a$ from product of Equations 29 and 26.

(17) Calculate the height of a transfer unit, $HTU_{oc} = U_c/K_{oc}a$

(18) Calculate the packing height Z (Equation 1)

Redistribution in packed liquid-liquid extractors appears to be very important. In a recent study (1), the overall mass transfer efficiency was observed to be dependent on the frequency of redistribution packed as shown in Figure 5. The decreased efficiency with increased bed height is likely due to phase maldistribution. There is a definite need for more future research in this area. Based on our current thinking it is recommended that packed beds be redistributed every ten feet.

CONCLUSIONS

As in distillation, structured packings offer advantages in capacity and mass transfer efficiency relative to other nonproprietary devices such as sieve trays and sprays. Structured packings are also very amenable to mechanistic modelling because of their ordered geometry within the extraction column. In order to optimize their design in liquid extraction service, one must be aware of the mechanisms controlling the process. Drop diameter, drop velocity, phase mass transfer resistance, fluid mixing, and contactor geometry must all be considered.

NOMENCLATURE

a	Interfacial area, cm^2/cm^3
a_p	Packing surface, cm^2/cm^3
C	Concentration, g/cm^3
C_c	Concentration of the continuous phase
C_c*	Equilibrium concentration of solute in the continuous phase, g/cm^3
$C_{c,i}$	Interfacial continuous-phase concentration of solute, g/cm^3
C_d	Concentration of the dispersed phase
$C_{d,i}$	Interfacial dispersed-phase concentration of solute, g/cm^3
C_D	Drag coefficient
$C*$	Equilibrium concentration
d_{vs}	Sauter mean drop diameter, cm
D_c	Diffusion coefficient of solute into the continuous phase, cm^2/s
D_d	Diffusion coefficient of solute into the dispersed phase, cm^2/s
F	Force, $g\ cm/s^2$
g	Gravitational constant, cm/s^2
HETS	Height equivalent to a theoretical stage
HTU_{oc}	Height of an overall transfer unit based on the continuous phase, cm
k_c	Continuous-phase film mass transfer coefficient, cm/s

k_d	Dispersed-phase film mass transfer coefficient, cm/s
K_{oc}	Overall continuous-phase mass transfer coefficient, cm/s
$K_{oc}a$	Continuous-phase volumetric mass transfer coefficient
m_{dc}	Distribution coefficient, dC_d^*/dC_c
N_t	Total mass flux, g/cm^2-s
Q_c	Volumetric flow rate of the continuous phase, cm^3/s
Re_c	Continuous phase Reynolds number
Re_d	Dispersed-phase Reynolds number
Sc_c	Continuous-phase Schmidt number, $\mu_c/\rho_c D_c$
Sc_c	Dispersed-phase Schmidt number, $\mu_d/\rho_d D_d$
U_{drop}	Drop velocity, cm/s
U_c	Superficial continuous velocity, cm/s
U_d	Superficial dispersed velocity, cm/s
U_{cf}	Continuous-phase flooding velocity, cm/s
U_{df}	Dispersed-phase flooding velocity, cm/s
U_{ic}	Interstitial velocity of the continuous phase
U_s	Slip velocity, cm/s
U_{so}	Characteristic slip velocity, cm/s
Z	Contacting height, cm

Greek Symbols

β_f	Fraction of flood
ε	Void fraction of the column ($\varepsilon = 1.0$ for sprays and sieve trays)
ϕ_d	Dispersed-phase holdup
Φ	Criteria for determining dispersed-phase mass transfer model
ρ_c	Continuous-phase density, g/cm^3
ρ_d	Dispersed-phase density, g/cm^3
$\Delta\rho$	Density difference, g/cm^3
μ_c	Continuous-phase viscosity, cp
μ_d	Dispersed-phase viscosity, cp
μ_w	Aqueous viscosity, 0.9 cp
σ	Interfacial tension, dynes/cm
λ	Extraction factor, $m_{dc}(U_d/U_c)$
ζ	Tortuosity factor, $a_p d_{vs}/2$

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