Study of Drop Size Distribution in RTL Contactor

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ABSTRACT

The present work studied the drop size distribution in the RTL contactor using two liquid-liquid systems, xylene-acetone-water and kerosene-acetone-water. Process variables studied were: rpm (10-50 min⁻¹), continuous phase flow rate (4-12 l/h), dispersed phase flow rate (4-12 l/h), and concentration (0.1-0.5 mole/l). It was found that Sauter mean drop diameter (d_{32}) decreased with increasing rpm and continuous phase flow rate, and decreasing dispersed phase flow rate and concentration. An empirical correlation, with a correlation coefficient equal to (92.6%), relating d_{32} with process variables was developed having the form:

$$\frac{d_{32}}{R} = 9.845 * 10^{-3} (\Phi)^{0.0627} (We)^{-0.367} (Re)^{0.228} (\frac{Q_d}{Q_c})^{0.43}$$

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KEYWORDS

Drop size distribution, RTL contactor, Liquid extraction.

INTRODUCTION

The separation of the constituents of a homogeneous liquid mixture is a problem frequently encountered in the chemical processing industry. Liquid-liquid extraction has been emerging as a very important method for separating such liquid mixtures. The addition of a solvent in this process corresponds to the addition of heat in the case of separation by distillation. The solvent used in the extraction process should be immiscible or partially miscible with one of the components of the mixture in order to facilitate the separation of liquid phases [Hartland 1970, Treybal 1981].

The case for the use of liquid-liquid extraction will depend upon its either accomplishing a separation that cannot be achieved by other operations such as: distillation, evaporation, and crystallization, or effecting the separation more economically. Liquid-liquid extraction is now being adopted as a more economic alternative to other separation processes and has found immense applications in the separation of [Benitez 2002]:

- 1. Solutions of components having low relative volatility, especially when vacuum distillation is expensive.
- 2. Solutions of close boiling and azeotrope forming components.
- 3. Dissolved solute when evaporation may be impractical.
- 4. Solutions of heat sensitive components such as antibiotics.
- 5. Components of differing chemical type whose boiling points may overlap as in the case of petroleum hydrocarbons.

The RTL (Formerly Graesser Raining Bucket) contactor consists of a single rotor operating on a substantially horizontal axis in a cylindrical stator. The rotor comprises a series of circular baffles, between which are mounted a number of cylindrical buckets, partly open in the direction of rotation. RTL contactor applied to many extraction processes, such as: pyrethrum refining, purification of herbicide products, and dearomatization of naphtha. Possibilities exist for the applications of the RTL contactor to systems containing a solid phases; thus apparatus has been used for the treatment of oil bearing sands, with the use of kerosene to remove bitumen as one product, leaving clean treated sand as the under flow [Tech, et al. 1983].

RTL contactor differs in some major respects from vertical type contactors. It has the operating advantages that:

- Density changes during extraction will not induce axial circulation since the axis is horizontal.
- 2- The RTL contactor recovers easily from any sudden shutdown since drainage of material from stage to stage cannot occur.

These factors in addition to its ability to handle easily emulsified systems, have contributed to its success over the past years. [Laddha and Degaleesan 1976, Tech, et al. 1983, Mark and Othmer 1998].

DROP SIZE DISTRIBUTION

The effects of drop size and size distribution on the performance of an extraction column are the most important hydrodynamic characteristics, because under steady operating conditions, drop size is related to the interfacial area by:

$$a_s = \frac{6\Phi}{d_{32}}$$

Where:

 Φ = Dispersed phase holdup.

$$d_{32} =$$
Sauter mean diameter $= \frac{\sum n_i d_i^3}{\sum n_i d_i^2}$

The prediction of interfacial area in agitated dispersions is of a considerable importance in heat and mass transfer operations.

Many investigators studied the effect of drop size distribution on the performance of different contactors. Drop size distribution is an important parameter as it defines the available interfacial area for mass transfer. No general correlation is available at present time to describe the drop size distribution in each contactor. Each investigator found a different distribution function suitable to describe his data [Lovick, et al. 2005]. No attempts have been done till now to study the effect of drop size distribution with mass transfer conditions on the performance of RTL contactor.

The following two tables shows some relations used to represent the drop size distribution with and without mass transfer conditions.

Hartland 1986].					
No	Correlation	Ref.			
1	$\frac{d_{32}}{\sqrt{\frac{\sigma}{\rho_c g}}} = 16.7(\frac{N^* R^2 \rho_c}{\mu_c})^{-0.3}(\frac{N^{*2} R}{g})^{-0.3}n^{-0.23}$	Kagan, et al. 1964			
2	$\frac{d_{32}}{R} = 0.037 \left(\frac{N^{*2}R^{3}\rho_{c}}{\sigma}\right)^{-0.6} \left(\frac{N^{*2}R}{g}\right)^{-0.27} \left(\frac{N^{*}R^{2}\rho_{c}}{\mu_{c}}\right)^{0.14} \\ \left(\frac{\mu_{d}}{\mu_{c}}\right)^{0.06} \left(\frac{D_{r}}{R}\right)^{0.875} \left(\frac{H}{w}\right)^{0.45} \left[1 + \frac{3.7}{\exp(0.008N^{*} + 0.25n)}\right]$	Fischer 1971			
3	$\frac{d_{32}}{D} = 0.62(\frac{N^2 D^3 \rho_c}{\sigma})^{-0.52} [1 + \frac{35}{n^{1.22}(\frac{N^2 D^3 \rho_c}{\sigma})^{0.5}}](1 + 2\varepsilon)$	Fischer 1973			
4	$\frac{d_{32}}{R} = 4.7 * 10^{17} (\frac{N^* R^2 \rho_c}{\mu_c})^{-3.33} (\frac{\mu_d}{\mu_c})^{0.23} (\frac{N^* R \mu_c}{\sigma})^2 \varepsilon^{0.225} \exp(0.4\frac{n}{n_t})$	Mumford and Al- Hemiri 1974			
5	$\frac{d_{32}}{R} = 0.17 \left(\frac{N^{*2} R^3 \rho_c}{\sigma}\right)^{-1.2} \left(\frac{N^{*} R^2 \rho_c}{\mu_c}\right)^{0.6}$	Mar, et al. 1975, 1978			
6	$\frac{d_{32}}{R} = 0.3(\frac{N^{*2}R^{3}\rho_{c}}{\sigma})^{-0.6}(\frac{N^{*2}R}{g})^{-0.27}(\frac{N^{*}R^{2}\rho_{c}}{\mu_{c}})^{0.14}$	Mersmann 1980			

 Table (1). Published correlation of drop size without mass transfer [Kumar and

 Hortland 1086]

Table (2). Published correlations of drop size under mass transfer conditions [Kumarand Hartland 1986].

No.	Correlation	Reference
1	$d_{32} = [1.04(\frac{R}{D_r} - 0.66) + 0.06]N^{*-2.2}$	Spathe and Weiss 1972
2	$\frac{d_{32}}{D_r} = 108*10^{-5} \left(\frac{N^* R^2 \rho_c}{\mu_c}\right)^{2.15} \left(\frac{N^{*2} R}{g}\right)^{1.05} \left(\frac{N^{*2} R^3 \rho_c}{\sigma}\right)^{-2.7} \left(\frac{U_d}{U_c}\right) \left(\frac{\sigma}{U_c \mu_c}\right)^{-0.27} \left(\frac{\mu_d}{\mu_c}\right)^{-1.93} \left(\frac{\rho_d}{\rho_c}\right)^{0.77} n^{-0.35}$	Magiera and Zadlo 1977
3	$\frac{d_{32}}{D_r} = 1.43 \left[\frac{U_d (1-\varepsilon)}{U_c \varepsilon}\right]^{0.45} \left[\frac{N^* R(1-\varepsilon)}{U_c}\right]^{-0.565} \left[\frac{\rho_c H U_c}{\mu_c (1-\varepsilon)}\right]^{-0.117}$	Blazej, et al. 1978
4	$\frac{d_{32}}{R} = 1.48 \left[\frac{U_d H \rho_c}{\varepsilon \mu_c}\right]^{-0.23} \left(\frac{N^{*2} R^3 \rho_c}{\sigma}\right)^{-0.004} \left(\frac{U_d^2}{\varepsilon^2 g D_r}\right)^{0.44} \\ \left(\frac{\Delta \rho}{\rho_c}\right)^{-0.57} \left(\frac{H}{D_r D_s}\right)^{-0.24} \left(\frac{n}{n_t}\right)^{-0.07}$	Jeffreys, et al. 1981

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$$\frac{d_{23}}{R} = 1.26(\frac{N^{*2}R^{3}\rho_{c}}{\sigma})^{-0.52}[\frac{36}{n^{1.22}(\frac{N^{*2}R^{3}\rho_{c}}{\sigma})^{0.5}}]$$
 Hartung and Weiss 1982

Shinnar and Church [1960] investigated the predication of drop size in agitated dispersions. The average drop diameter was calculated for concentrated liquid-liquid distribution in agitated tanks for various liquids by the following empirical equation derived by Rodger, et al. [1956].

$$d_{av} = \frac{R}{k} \left(\frac{R^3 N^{*2} \rho_c}{\sigma}\right)^{-0.36} \left[\frac{R^k}{D_r} \left(\frac{v_d}{v_c}\right)^{0.2} \left(\frac{t}{t_o}\right)^{\frac{1}{6}} \exp(3.6\frac{\Delta\rho}{\rho_c}) 0.71 \exp(0.35\frac{D_r}{D_{ro}})\right]^{-1}$$

(1)

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Hinze [1955] has proposed an equation for the maximum stable drop size, based on the assumption of an isotropic homogeneous turbulence in liquid-liquid systems with mass transfer:

$$d_{\max} = 0.725 (\frac{g_c \sigma}{\rho_c})^{\frac{3}{5}} \varepsilon^{-\frac{2}{5}}$$
(2)

Strand, et al. [1962] studied the performance of rotating disc contactors under mass transfer conditions. Some attempts to measure (d_{max}) have been made. The authors couldn't obtain enough information to generalize, but it can be said that such data will be more difficult to interpret, especially if rates of drop breakup or coalescence are sensitively affected by solute concentration or by the direction of transfer. They suggested that a mechanism for predicting (d) or (d_{max}) during mass transfer will be difficult to devise.

Olney [1964] investigated the prediction of drop size distribution in a countercurrent rotating disc contactor for five liquid-liquid systems. The study presented an interpretation of drop size distribution data in a contactor for which a fairly complete analysis of drop dynamics and mass transfer processes were obtained based on the simplified (average drop) concept. Olney showed that rotor speed have a marked effect on the average or characteristic drop size but not much effect on the parameters of the size distributions. For example, the data for white oil dispersed in water showed that the volume median drop size (d_{50}) decreased from (3.1-0.8 mm) as rotor speed is increased from (650-1100 rev/min) but the width of the size distribution changed only from (4.5 to 4.2).

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Effect of holdup on the size distribution at a given rotor speed, as the holdup increased the increase in drop sizes over the whole spectrum may be caused solely by higher drop interaction rates due to smaller inter particle distances.

Olney showed that all his experimental data of the five systems were obeying the upper-limit distribution proposed by Mugele and Evans (1951).

Coulaloglou and Tavlarides [1976] studied the drop size distributions and coalescence frequencies of liquid-liquid distributions in flow vessels. Drop size distribution measurements in the circulation and impeller regions and dispersed phase coalescence frequencies measurements were made co-currently for the turbulently agitated dispersion of kerosene – dichlorobenzene (d) - in water (c), in a 12 liter flow vessel. The ranges of operating conditions studied were dispersed phase holdup (0.025-0.15), impeller speed (190-310 rev\min) and nominal residence time of (10min). The Sauter mean diameter was correlated by the following relation:

$$\frac{d_{32}}{R} = 0.081(1 + 4.47\Phi)(We_{crit})^{-0.6}$$
(3)

Magiera, et al. [1976] investigate the hydrodynamics of rotating disc contactor. They showed that Sauter mean diameter is calculated from:

$$d_{32} = [1.045(\frac{R}{D_r} - 0.66) + 0.06]N^{*2.2}$$
(4)

Many studies have been done on the bubble size distribution in different contactors [Alves, et al. 2002, Mouza, et al. 2005, and Lage 1999]. Martinez, et al. [1999] studied the breakup frequency of air bubble in a fully turbulent flow. They found that the diameter for which breakup frequency will be a maximum depends on:

$$d_{\max} = 1.63 \left(\frac{12\sigma}{8.2\rho}\right)^{\frac{3}{5}} \varepsilon^{-\frac{2}{5}}$$
(5)

EXPERIMENTAL WORK:

Liquid-Liquid Systems: -

Physical properties of the liquid-liquid systems employed are summarized in table (3). The selection of these two systems is based in general on:

- 1. Availability and cost.
- 2. Selectivity.

- 3. Difference in physical properties.
- 4. Corrosivity.

The systems used were:

- 1. Water (c)-acetone (s)-xylene (d).
- 2. Water (c)-acetone (s)-kerosene (d).

System	Temp	Density	y g/cm ³	Visco g/cm	osity .sec	Diffu cm ²	sivity /sec	Interfacia l tension dyne/cm	Distributi on coefficient
xylene- acetone-water	25	Disp. 0.860	Cont. 0.997	Disp. 0.610	Cont. 1.020	Disp. 0.389	Cont 0.109	28.01	0.438
kerosene- acetone-water	21	0.795	0.998	1.123	1.050	0.227	0.109	39.9	0.905

Table (3) Physical properties of the studied systems.

Description of Equipment: -

A (0.1 m) diameter, (1 m) long Graesser raining bucket contactor supplied by Corning Ltd., England, was employed. The contactor consists of a horizontal cylindrical shell with a compartment rotor which is made up of a number of circular plates which have "C-shape" buckets mounted near the outer edge between each pair of plates.

The contactor is capable of handling a total phase throughput of (12 l/h) and it consist of (36) compartments, each of (0.025 m) long and consists of (6) buckets, each bucket is (0.025 m) in diameter.

The two phases flow counter-currently along the shell from compartment to another through a peripheral gap between the rotor and the shell. A short disengaging zone is provided at each end of the unit to ensure complete separation of the phase. The heavy phase, which was aqueous phase, enters and leaves the contactor at points just below the rotor shaft, while the light (organic) phase enters and leaves at points above the rotor shaft. A (1/3 hp) AC motor drives the rotor shaft. The drive shaft of the motor was coupled to the contactor shaft via a gear box to select the required rpm (0-50).

Six (0.01 m³) QVF glass spherical vessels were used as storage reservoirs and were mounted on a special vessel support. Two Stuart-Turner centrifugal pumps, type

number 12, capable of handling (720-750 gph) against (10-45 ft) head of water were used to transfer the liquids.

The pumps were made of stainless steel and the pump glands were especially adapted for use with solvents.

Flow rates were measured by independently calibrated rotameters. The range of flow of each rotameter is (2.4-30 L/h) of water at 20 0 C.

MEASUREMENTS OF SOLUTE CONCENTRATION: -

Concentrations of solute in organic phase and aqueous phase were measured by UV-visible spectrophotometer (Shimadzu UV-160).

EXPERIMENTAL PROCEDURE: -

The dispersed phase solution was made up to the required concentration. The contactor filled with the continuous phase, and then the dispersed phase was introduced into the contactor. Flow rates were adjusted to the required value until the dispersed phase made up to (50%) holdup. The continuous phase flow and the agitator were adjusted to the required values.

Pictures were taken at several compartments of the contactor, namely compartment [6, 11, 16, 21, 26, and 31]. After steady state was reached, pictures were taken.

LG web camera, version LG Lic-100, connected to P4 computer, which have the ability for recording video or pictures, used for picturing. The camera has the ability to record (33 frames per second).

Lighting was provided by two (1000, 250 watt) photoflood lamps placed directly behind the contactor.

Pictures were taken through the glass shell of the contactor, and the area pictured was (0.025 m) in width, bracketing one compartment, and (0.03 m) in length. And since the object is very close to the contactor wall distortion due to curvature is therefore negligible.

(800) to (1500) pictures were taken for each experiment (130 to 250 pictures in each compartment). Drop size measurements were taken from pictures using picasa programmed in P4 computer to calculate the drop size, (15-35) picture were analyzed

for each compartment. Finally, samples from liquid vessels were taken and analyzed by UV-spectrophotometer to obtain the concentrations of solute.

PROCESS VARIABLES: -

Table (5) shows the range of operating conditions studied in the present research: -

	Variable	Range
X_1	Rotation of speed	10-50 rpm
X_2	Continuous phase flow rate	4-12 l/h
X ₃	Dispersed phase flow rate	4-12 l/h
X_4	Concentration of dispersed phase	0.1-0.5 mole/l

Table (5) operating conditions studied

EMPIRICAL MODEL OF SAUTER MEAN DIAMETER: -

The effect of operating conditions, physical properties, and contactor geometry on the Sauter mean diameter of each experiment were correlated using dimensionless analysis (Buckingham theorem).

d₃₂ is assumed to be a function of the following variables:

$$d_{32} = f(Q_d, Q_c, \Phi, \mu_d, \mu_c, \rho_d, \rho_c, N^*, \sigma, R)$$
(6)

$$d_{32} = i * R * (\Phi)^{i_1} (\frac{\rho_c N^{*2} R^3}{\sigma})^{i_2} (\frac{Q_d}{Q_c})^{i_3} (\frac{\mu_d}{\mu_c})^{i_4} (\frac{\Delta \rho}{\rho_c})^{i_5} (\frac{N^* R^2 \rho_c}{\mu_c})^{i_6}$$

Using STATISTICA program, the constant (i) and the powers were found to be: $i = 9.845*10^{-3}$ $i_1 = 0.0627$ $i_2 = -0.367$ $i_3 = 0.4003$ $i_4 = 0.0723$ $i_5 = -0.00818$ $i_6 = 0.228$

Thus the final correlation obtained is:

$$\frac{d_{32}}{R} = 9.845 * 10^{-3} \, (\Phi)^{0.0627} (We)^{-0.367} (\text{Re})^{0.228} (\frac{Q_d}{Q_c})^{0.43} \tag{7}$$

Since modified We no. = $\frac{\rho_c N^{*^2} R^3}{\sigma}$ and modified Re no. = $\frac{N^* R^2 \rho_c}{\mu_c}$

This equation correlates the data with a correlation coefficient equal to (92.6%).

RESULTS AND DISCUSSIONS :

The Influence of Process Variables on Sauter Mean Drop Diameter (d₃₂): -

Variation of d_{32} along the axial distance of the contactor is shown in figures (1) for xylene-acetone-water system and (2) for kerosene-acetone-water system. The two figures showed that the d_{32} decreased with the axial distance from the dispersed phase inlet. This was because, in the inlet of the dispersed phase and due to the horizontal nature of the contactor the holdup is higher in compartment number one than that in the other compartments and decreased with the axial distance along the contactor and reached a minimum value at the inlet of the continuous phase. This was in agreement with Al-Hemiri and Kareem [1990], as the holdup increased the d_{32} increase.

EFFECT OF RPM: -

Figures (3) and (4) show the effect of rpm on d_{32} for the two systems xyleneacetone-water system and kerosene-acetone-water system respectively.

The two figures showed that increasing the rpm decreased d_{32} . This was because increasing the rpm increased drop breakup and lead to smaller drops. This was in agreement with Coulaloglou and Tavlarides [1976], Lovick, et al. [2005].

Equation (7) shows a good agreement with the experimental results as shown in the two figures.

EFFECT OF CONTINUOUS PHASE FLOW RATE: -

Figures (5) and (6) show the effect of continuous phase flow rate on the d_{32} for the two systems xylene-acetone-water system and kerosene-acetone-water system respectively.

Increasing the continuous phase flow rate decreased the d_{32} for the two systems. This was because an increase in continuous phase flow rate leads to more redispersion resulting in smaller drops. This was in contrary to the observations of Mumford [1970], who suggested that in agitated columns, the vertical contactors like the RDC, the drop size distribution was directly affected by the continuous phase flow rate, but it is in agreement with Al-Hemiri and Kaream [1990], and Kolb and Bart [2002]. ()

Equation (7) shows a good agreement with the experimental results as shown in the two figures.

EFFECT OF DISPERSED PHASE FLOW RATE:-

Figures (7) and (8) show the effect of dispersed phase flow rate on d_{32} for the two systems xylene-acetone-water and kerosene-acetone-water respectively.

Increasing the dispersed phase flow rate increased the d_{32} , this was because a larger amount of the dispersed phase enhance coalescence and cause an increase in the drop sizes. This was in agreement with Jeffreys, et al. [1981], and Al-Hemiri and Kareem [1990].

Equation (7) shows a good agreement with the experimental results as shown in the two figures.

EFFECT OF CONCENTRATION: -

Figures (9) and (10) show the effect of concentration on d_{32} for the two systems xylene-acetone-water and kerosene-acetone-water respectively.

For both systems used increasing the concentration increased the d_{32} this is because the transfer is from the dispersed phase to the continuous phase, which enhances the drop size to be larger due to the Marangoni effect, This was in agreement with Kumar and Hartland [1986] and Bratsun and Dewitt [2004].



Fig. (1). Sauter mean diameter along the axial contactor length experiment (8) xylene-acetone-water system.



Fig. (2). Sauter mean diameter along the axial contactor length experiment (20) kerosene-acetone-water system.



Fig. (3). Effect of rpm on the Sauter mean diameter for xylene-acetone-water system.



Fig. (4). Effect of rpm on the Sauter mean diameter for kerosene-acetone-water system.



Fig. (5). Effect of continuous phase flow rate on the Sauter mean diameter for xylene-acetone-water system.



Fig. (6). Effect of continuous phase flow rate on the Sauter mean diameter for kerosene-acetone-water system.



Fig. (7). Effect of dispersed phase flow rate on the Sauter mean diameter for xylene-acetone-water system.



Fig. (8). Effect of dispersed phase flow rate on the Sauter mean diameter for kerosene-acetone-water system.



Fig. (9). Effect of concentration on the Sauter mean diameter for xylene-acetone-water system.



Fig. (10). Effect of concentration on the Sauter mean diameter for kerosene-acetone-water system.

CONCLUSIONS:-

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* The Sauter mean drop size (d_{32}) decreased with the axial distance along the contactor from the point of the inlet of the dispersed phase.

* An empirical correlation was correlated between d_{32} and operating conditions, contactor geometry, and physical properties of the studied system, expressed as a function of dimensionless groups with a correlation coefficient of (92.6%).

* The d₃₂ was found to decrease with:

- Increasing rotation of speed (rpm).
- Increasing continuous phase flow rate.
- Decreasing dispersed phase flow rate.

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Symbol	Definition	Units
А	Interfacial mass transfer area	cm^2
as	Specific interfacial mass transfer area	cm^2/cm^3
D	diffusivity	cm^2/s
D _r	Column diameter	cm
d ₃₂	Sauter mean drop diameter	cm
g	Acceleration of gravity	cm/s ²
Н	Width of contactor compartment	cm
\mathbf{h}_{m}	Height of mixing compartment	cm
Ko	Overall mass transfer coefficient	cm/s
k _c	Individual mass transfer coefficient of continuous phase	cm/s
K _{oc}	Overall mass transfer coefficient based on continuous	cm/s
	phase	
K _{od}	Overall mass transfer coefficient based on dispersed	cm/s
	phase	
k _d	Individual mass transfer coefficient of dispersed phase	cm/s
L	Length of contactor	cm
m	Distribution coefficient	
n	Compartment number	
\mathbf{N}^{*}	Rotor speed	\min^{-1}
Q	Volumetric phase flow rate	l/h
R	Impeller (rotor) diameter	cm
$\mathbf{S}_{\mathbf{f}}$	Superficial flow velocity	$cm^3/cm^2.s$
U	Superficial phase velocity	cm/s
U_k	Characteristic velocity	cm/s

NOMENCLATURE:-

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Ut	Terminal velocity	cm/s
V	Volume of contactor	1
X _{1,2,3,4}	Process variables	

GREEK SYMBOLS:

Symbol	Definition	Units
3	Dissipation energy per unit mass	erg/g.s
μ	viscosity	g/cm.s
ν	Kinamatic viscosity	cm^2/s
ρ	density	g/cm ³
σ	Interfacial tension	dyne/cm
Φ	Dispersed phase holdup fraction	

SUBSCRIPT:

Symbol	Definition	1
с	Continuous phase	
d	Dispersed phase	
max	Maximum	
min	Minimum	
crit	Critical	