

Dispersion Parameters and Effect of Impeller Speed, Holdup and Volume Fraction of Dispersed Phase on Separation Efficiency, Mass Transfer Coefficient of Dispersed Phase and Distribution Coefficient on Mixer-Settler Set

Saadat Gharehbagh, Fatemeh*⁺; Mousavian, Sayed Mohammad Ali

Faculty of Chemical Engineering, University of Tehran, Tehran, I.R. IRAN

ABSTRACT: An experimental study has been conducted on the hydrodynamics of a stage mixer-settler to obtain an appropriate design. In this paper several tests were performed to investigate full factorial design of experiments. Since each test was repeated seven times, the repeatability of the test was confirmed ($P=1$ bar and $T=25$ °C). Sauter diameter was determined by photographing both the mixer and settler in each test. The Sauter diameter was compared with the Calderbank model; finally, a model was suggested. Holdup quantity was measured by a vacuum pump at the end of each test. Another aim of this paper is the evaluation of separation mechanism under different impeller speeds and volume fractions. The effect of impeller speed in constant ratio of phases flow rate, and hold up are evaluated, and the effect of phase flow rate ratios in constant impeller speed on extraction efficiency, mass transfer coefficient and distribution coefficient were determined.

KEY WORDS: Mixer-Settler, Drop size, Dispersed phase hold up, Extraction efficiency, Mass transfer coefficient, Distribution.

INTRODUCTION

Zirconium is one of the abundant elements (162 ppm) and is widely distributed in the Earth's crust. Most of the zirconium is used as compounds for the ceramic industry, refractory's, glazes, enamels, foundry mold and abrasive grits, and compounds for electrical ceramics. The incorporation of zirconium oxide in glass significantly increases its resistance to alkali. Zirconium metal is used almost exclusively for cladding uranium fuel elements for nuclear power plants. Another significant use is in photo

flash bulbs. Some chemical processing industries use zirconium metal for corrosion-resistant vessels and piping, particularly for withstanding hydrochloric and sulfuric acid (Rajmane et al, 2006) [11].

Solvent extraction is well known as an effective method for the separation of lanthanides on the industrial scale from the acidic leaching solution of raw materials. However, even in the solvent extraction process, a large number of stages in a series of mixer-settlers are required

* To whom correspondence should be addressed.

+ E-mail: soroorus@gmail.com

1021-9986/10/3/

16/\$/3.60

for the preparation of high-purity products, because the chemical properties of adjacent elements are very similar. Various kinds of extraction columns have been studied extensively for applying liquid-liquid extraction. However, the mixer-settler extraction column showed better characteristics than the other types of extraction column. In our previous work, a mixer-settler extraction column having a vertical arrangement of mixer and a horizontal arrangement of settler in every stage was proposed, and it was shown that both the throughput and the stage efficiency increase when increasing the agitation speed. Another advantage of this mixer-settler extraction column is the independence of each stage of the column. Since the partition between stages is more complete than those in the other extraction columns, the hydrodynamics of one stage is not affected by the neighboring stages (Giraldo-Zuniga *et al*, 2006) [8].

In this article, a detailed experimental study has been therefore carried out on the hydrodynamics of a stage mixer-settler in a pilot-scale to obtain design criteria. The work included investigation of (a) dispersion characteristics in the mixer: drop-size distribution, mean drop size and dispersion phase hold up (b) separation mechanism of phases.

This article aims at drawing the drop distribution graph and dispersion depth measurement in different conditions of the mixer, and ratio of phases flow rate. Furthermore, measuring the machine's performance volume and separation index, along with the germ transfer index and the diffuse hold up phase are among other goals that this article seeks to achieve. Besides, the correlation of these parameters with one another, and acquiring the optimum point of extraction is another conducted operation. Coming up with the proposed model for this specific system is another objective to be accomplished in this article.

The hydrodynamic behavior and the mass transfer characteristics of the column have been analyzed experimentally. (Rajmane *et al*, 2006) [11].

The methods commonly employed are based either on the residence time of the phases or the thickness of the dispersion (or wedge) in the settler; however, important design parameters have been ignored; for example, the geometry of the settler as related to the mixer design, coalescence characteristics inside the settler, and phase stability to ensure minimum entrainment. Industrial

Table 1: Physical properties of system at P=1bar and t=25°.

Physical Properties	Tri-butyle phosphate(TBP) 60%, Kerosene instead organic phase and HNO ₃ (3.5M), Zirconium OxyChloride(12g/L)
μ_d (cp)	2.58
μ_c (cp)	0.958
ρ_d (g/cm ³)	0.907
ρ_c (g/cm ³)	1.204
σ (dyne/cm)	32.16
Dc (m ² /s)	4.251×10^{-8}
Dd (m ² /s)	4.60×10^{-8}

settlers are usually operated in a mode whereby the wedge extends across the entire length of the settler, thereby enhancing the probability of mutual phase entrainment. Industrial equipment seldom operates under steady-state conditions. Process fluctuations typically occur, and this often results in severe phase entrainment. Furthermore, the mixers are usually over designed through lack of data relating to volumetric capacity. Evaluation of a design procedure for a mixer-settler unit requires knowledge of the hydrodynamic systems. (Giraldo-Zuniga *et al*, 2005) [6].

Mass transfer flux is determined by molecular diffusion with first Fick law near the liquid-liquid interface. (Schloman 2005) [15].

EXPERIMENTAL SECTION

Materials

Chemical systems used in this set contain TBP 60%, kerosene for the organic phase, and HNO₃ (3.5M), zirconium oxychloride (12g/L) for the organic phase. Organic phase is dispersed and zirconium is solute, so it transfers between two phases.

Physical properties of the system are given in Table 1.

Methods

Experiments were designed according to the full factorial method, which is shown in Table 2.

Several tests did with high reliability, determined by each test recurring seven times and the results being very close to each other. Sauter diameter in each test was determined by photographing both the mixer and settler, and holdup

Table 2: Effective factors in experimental section at $P = 1\text{bar}$ and $t = 25^\circ\text{C}$.

1-Volume fraction of TBP to kerosene in organic phase	One case: volume fraction of TBP to kerosene is 60%
2- Concentration of nitric acid	One case: 3.5 M
3- Concentration of zirconium oxy chloride	One case: 12 g/L
4-Flow rate of organic phase to aqueous phase($V_d/V_c=k$, V_d, V_c : mL/min)	Three case:
	1. $k > 1$
	2. $k < 1$
	3. $k = 1$
5-Impeller speed(rpm)	Four case:
	750
	800
	900
	1 000

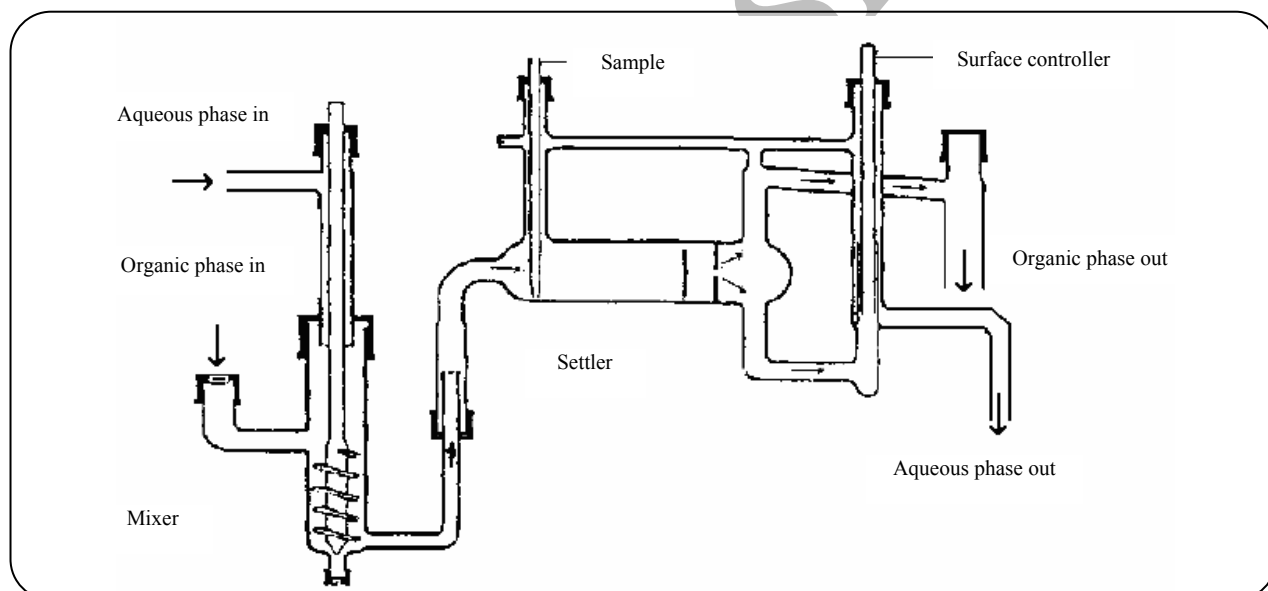


Fig. 1: Flow diagram of a stage of extraction system in a Mixer - Settler unit.

quantity is measured by a vacuum pump at the end of each test, so the ratio of organic phase quantity to quantity of both organic and aqueous phases was retained.

Settler characteristics were studied by photographing a wedge by means of mirrors inclined at 45° above and below the settler. This procedure enabled the upper and lower surfaces of the wedge to be viewed and photographed. Wedge dimensions were measured and photographed over a range of phase flow rates and energy input to the mixer. Baffle positions of 125 mm and 145 mm from the phase input were used.

At the start of each run the mixers and settlers were filled with two liquids until the height of liquid in the mixer was equal to the mixer diameter and the interface was at the mid-position in the settler. After setting of agitator speed, the feed and solvent flow rates were adjusted to give the required ratio of $V_c:V_d$.

Devices

The continuous countercurrent mixer-settler was incorporated (Fig. 1). The mixing vessel was provided with a turbine, and the settler was provided with two

Table 3: Parameters of Mixer-Settler unit.

Parameters	Definition
Constructed	Glassware
Number of stage	10
Dimension of system (cm)	150×125×100
Capacity of mixer(mL)	100
Diameter of mixing vessel(mm)	37
Capacity of settler(mL)	250
Settler diameter(mm)	50
Length of settler(mm)	125
Length of vessel after baffle(settler)(mm)	20
Length of separation vessel(mm)	45
Mixer diameter(mm)	24
Mixer gum(mm)	15
Speed(rpm)	1-1 000
Flow rate of pump(pulls/min)	0-120

vertical baffles. Phase separation occurred in horizontal, cylindrical settlers constructed of industrial glassware. The ratio of the mixed volume to the available settler volume was approximately 1:2.5. The ratio of impeller to vessel diameter (D/DT) was taken as 0.34, consistent with standard tank configurations. Parameters of the device are given in Table 3.

Two vertical disk baffles in each settler were installed opposite the inlet.

The "ideal" liquid-liquid, mutually saturated, system TBP 60%, kerosene- HNO_3 , 3.5M was used in the investigation. In most runs, HNO_3 was the continuous phase. Direct photography was used for drop-size measurement.

A digital camera (10.0 megapixel with 12 x optical zoom) was used for this experiment. The Sauter diameter in each test was determined by photographing (angle of 45°) of both the mixer and settler, while the dispersion wedge was determined by photographing of the settler at a 45° angle, and Evaluation of the separation mechanism at different impeller speeds and volume fractions was another aim of this paper, and photos analyzed in auto cad (2009) software.

All the materials are supplied from Fulka company.

There were several effective items in experimental section and data exhibition such as instrument error:

Calibration of the instrument was not carried out or was faulty. Consequently accuracy and precision were affected, personal error: observer made inaccurate observations; these errors could be overcome by taking an average based on several measurements; moreover, the problem of personal bias resulting from poor observational habits which produced a consistent observational error, sampling errors: replication of experiments also reduced errors, estimated errors in set, software, camera,....

At the start of each run the mixers and settlers are filled with two liquids until the height of liquid in the mixer is equal to the mixer diameter and the interface is at the mid-position in the settler. After setting of agitator speed, the feed and solvent flow rates are adjusted to give the required ratio of $V_d:V_c$.

THEORY

Drop Size

Initially, the Sauter mean droplet diameter was determined as a function of time in order to determine the time for dispersion equilibrium. In all cases, 10 min. was found to be sufficient for achievement of equilibrium, resulting in a homogeneous dispersion with a relatively small drop size distribution. The critical speed for substantially uniform dispersion was approximately 700 rpm for the system. The Sauter mean drop diameter was calculated from the usual expression as Eq. (1) (Chakra Borty et al, 2003) [2].

$$d_{32} = \frac{\sum_{j=1}^n n_j d_j^3}{\sum_{j=1}^n n_j d_j^2} \quad (1)$$

There are two types of liquid-liquid extraction.

a) Primary dispersion: in this dispersion, drop size is more than 100 microns.

b) Secondary dispersion: in this dispersion, drop size is less than 1 micron (Chouai et al, 2000 [3]; Ruivo et al, 2006 [12]).

Mass transfer coefficients

Usually, chemical engineers use mass transfer flux with both mass transfer coefficient and concentration of solute in each phase.

Measuring of solute concentration in a liquid-liquid interface is too difficult, so we prefer to use overall mass transfer coefficient, Eq. (2)

Table 4: Experimental results of experience (Sauter diameter and Dispersed phase hold up).

N(rpm)	Vd(mL/min)	Vc(mL/min)	Hold up	Vd/Vc	Experience d32(mm)	Suggested model	Error of Suggested Model	Calderbank	Error of Calderbank model
750	59	45	0.50	1.31	0.26 000	0.25 010	0.00 010	0.22 452	0.00 126
800	59	45	0.48	1.31	0.23 000	0.22 293	0.00 005	0.20 237	0.00 076
900	59	45	0.46	1.31	0.2 000	0.18 613	0.00 019	0.17 099	0.00 084
1 000	59	45	0.40	1.31	0.17 000	0.14 443	0.00 065	0.13 824	0.00 101
750	50	50	0.42	1.00	0.19 000	0.21 320	0.00 054	0.20 110	0.00 012
800	50	50	0.41	1.00	0.18 000	0.19 304	0.00 017	0.18 340	0.00 001
900	50	50	0.40	1.00	0.17 000	0.16 389	0.00 004	0.15 687	0.00 017
1 000	50	50	0.38	1.00	0.15 000	0.13 789	0.00 015	0.13 409	0.00 025
750	42	53	0.36	0.79	0.15 000	0.18 522	0.00 126	0.18 352	0.00 112
800	42	53	0.32	0.79	0.14 000	0.15 462	0.00 021	0.15 901	0.00 036
900	42	53	0.28	0.79	0.13 000	0.11 942	0.00 011	0.12 864	0.00 000
1 000	42	53	0.26	0.79	0.12 000	0.09 870	0.00 045	0.10 921	0.00126
Sum of least square error							0.00393		0.00 604

Mass transfer coefficient of dispersed phase is measured by Eq. (2)

$$\dot{m}_d(y_{out} - y_{in}) = K_d a(C_d^* - C_{d,out})V_d \quad (2)$$

Mass transfer efficiency

Evaluation of both mixer-settler efficiency and the effect of several operating factors in the devices are the main design parameters in this field.

Extraction efficiency helps us to optimize solvent quantity and impeller speed as well. In general, mass transfer efficiency is the function of several parameters such as impeller speed, solvent quantity, and hold up.

The relationship for determining extraction efficiency is submitted in Eq. (3).

$$E_{ext} = \frac{X_{in} - X_{out}}{X_{in} - X^*} \quad (3)$$

Generally, with Equation (3) and concentration data, we can discover and evaluate efficiency (Staszak & Prochaska 2006) [17].

Distribution coefficients

The ratio of the total analytical concentration of a solute in the extract (regardless of its chemical form) equals its total analytical concentration in the other phase.

The terms distribution coefficient, extraction coefficient, and where appropriate, scrubbing coefficient and stripping coefficient are widely used but are not recommended here. If they should be used in a given situation, the term "ratio" is preferable to "coefficient". In equations relating to aqueous/organic systems, the organic phase concentration is, by convention, the numerator and the aqueous phase concentration is the denominator. In the case of stripping ratio the opposite convention is sometimes used but should be clearly specified.

Distribution coefficient is determined with Eq. (4) (Staszak & Prochaska 2006) [17].

$$D = \frac{[M]_{org}}{[M]_{aq}} \quad (4)$$

RESULTS AND DISCUSSION

Drop distribution

The study, in this paper, is about primary dispersion, as shown in Table 4.

Dispersed phase hold up is specified as the method in the Experimental Section, and the results of these experiments are shown in Table 4 and illustrated in Fig. 2. Here, variation of the Sauter diameter (d32) versus hold up under constant impeller speed is delineated. Another previously

studied proposition with economic and industrial significance is drop size distribution in different conditions, such as different impeller speeds and different ratios of organic phase flow rate to aqueous phase flow rate, so the optimum condition for extraction in experience system occurs (Saien *et al*, 2006) [14].

Drop size distribution curves are illustrated in Figs. 3 and 4. In these curves, ratio of number of droplets that have average contiguous Sauter diameter in a group to total population of droplets ($d \times n$)/ dn versus amount of Sauter diameter of this group, is charted.

The range of drop sizes observed in the mixer was mainly between 0.12 and 0.26 mm, the characteristic of those normally found in agitated aqueous organic systems. In all cases, some droplets were produced in the secondary dispersion size range of less than 0.1 mm, but these will create no difficulty in the settler because, at the used energy inputs, they were a small proportion of the dispersed phase. Drop size distributions were measured with a range of rotor speeds and phase flow rates. Figs. 3 & 4 are typical of the distribution curves obtained. Precise comparison is difficult since drop sizes are very much dependent on the geometrical configuration of the contactor (Dehkordi, 2002 [5]; Dehghani & Foster, 2001 [4]; Schmidt *et al*, 2006 [16]).

There are several models of droplet distribution shown in Table 5:

As shown in this Table, the Calderbank model with 4 blade paddles is the only model which is close to our proposed system in dispersed phase density, so it is used in experiments to produce comparison data for this model. In the end we suggest a model using the sum of least square method that is equal to this system.

Determination of suggested model Parameters for experimental system

Experimental values of d_{32} were compared with the correlation of Calderbank which is shown in Eq. 5.

$$\frac{d_{32}}{D} = 0.06(1 + 3.75\phi)(We)^{-0.6} \quad (5)$$

The difference in results is probably due to the different geometric configurations of the mixing systems used, and due to the presence of a settler in the present work, which creates a back pressure related to the wedge dimension; that is, the greater the hindrance, the larger

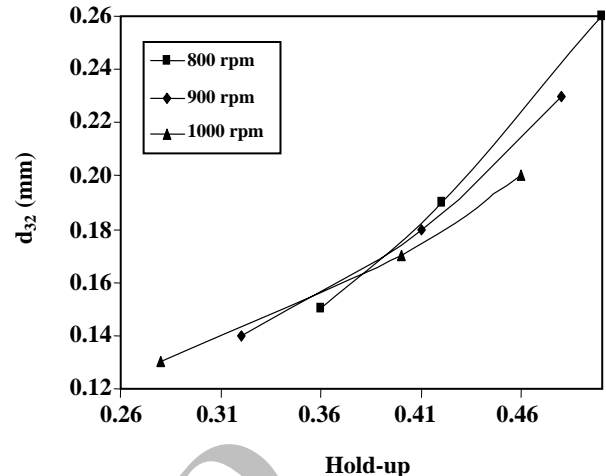


Fig. 2: Drop size (Sauter mean drop diameter) vs. holdup with different constant impeller speeds.

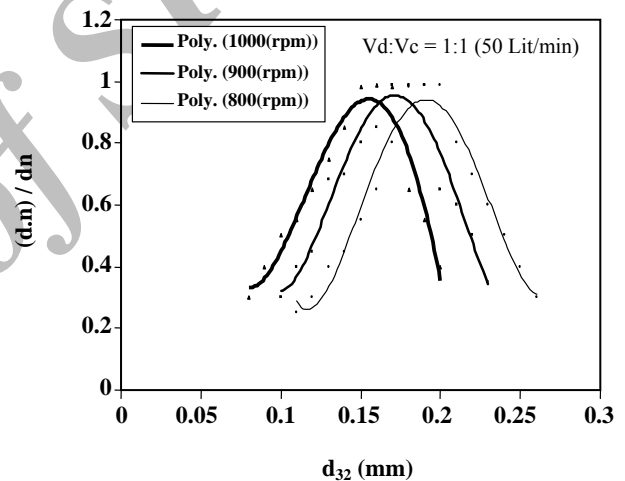


Fig. 3: Drop size distribution with impeller speed.

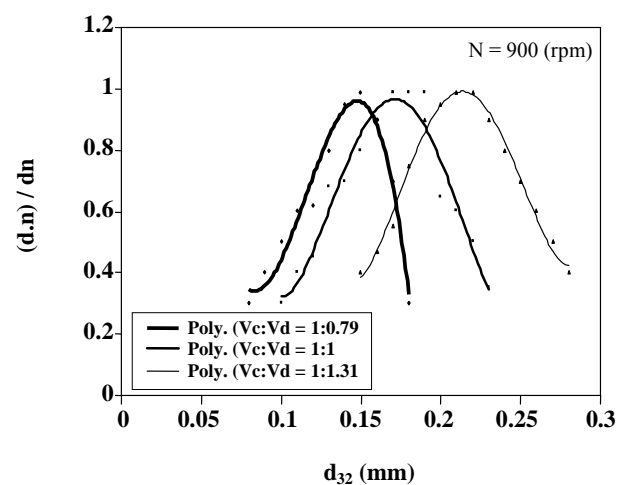


Fig. 4: Drop size distribution with volume fraction of droplets.

Table 5: Several models about droplet distribution.

Investigator	Correlation	pd (gr/ml)
Vermeulen, Calderbank, Sprow(a), Chen & Middleman	$d_{32} / D = C_1 (We)_T^{-0.6}$ $0.51 \leq C_1 \leq 0.53$	0.692-1.20
Shinar, Sprow	$d_{32} / D = C_2 \sigma D (We)_T^{-0.375}$ C2= experimentally determined	0.80
Sprow(a)	$d_{32} / D = C_3 (\rho_c \mu_c)^{-0.5} (\rho_c / \sigma D)^{-0.25} (We)_T^{-0.75}$ C3= experimentally determined	0.80
Calderbank	$d_{32} / D = 0.06(1 + 3.75\phi)(We)_T^{-0.6}$ (4bladepaddle) $d_{32} / D = 0.06(1 + 95\phi)(We)_T^{-0.6}$ (6bladepaddle)	0.87-1.59
Brown & Pitt(1)	$d_{32} / D = 0.051(1 + 3.14\phi)(We)_T^{-0.6}$	0.783-0.838
Mlynek & Resnik	$d_{32} / D = 0.058(1 + 5.4\phi)(We)_T^{-0.6}$	1.055
Present Work	$d_{32} / D = 0.081(1 + 4.47\phi)(We)_T^{-0.6}$	0.972
Brown & Pitt(2)	$d_{32}^{5/2} (\rho / \sigma) (e^{2/3} t_c / D_T) = c$ c= experimentally determined	0.783-0.838
Bouyatiotis & Thornton	$d_{32} = 5.38(\mu_c / \rho_c g) [(\rho / V_T)^3 g^3 / \rho_c^2 \mu_c g^4]^{-0.16}$ $[\rho_c^3 \sigma / \mu_c^4 g]^{-0.07} +$ $1.18\phi[\sigma^2 / \mu_c^2 g][\Delta\rho\gamma^3 / \mu_c^4 g]^{-0.62}[\Delta\rho / \rho_c]^{0.05}$	0.691-0.905

the back pressure. Under steady state conditions, momentum transfer in the mixer-settler interconnection enhances the level of turbulence in the mixer (Giraldo-Zuniga et al, 2005 [6]; Gottlieb et al, 2000 [9]).

A correlation can be derived from dimensional analysis of the physical properties of the system, operating conditions, and the impeller geometry, as given in Eq. (6).

It is assumed D , N , and σ are the agitator diameter, impeller speed, and surface tension, respectively, as described by Eq. (7). It is observed that d_{32} directly depends on dispersed phase hold up, as shown in Table 4. Therefore, a relationship for the Sauter diameter versus dimensionless parameters, such as dispersed phase hold up and Weber number, is proposed in Eq. 8 (Ruiz et al, 2002 [13]; Pinto et al, 2004 [10]; Ali et al, 2006 [1]).

$$d_{32} = f(N, V_d, V_c, \mu_d, \mu_c, \sigma_i, \rho_m, D) \quad (6)$$

$$We = \frac{D^3 N^2 d_{32}}{\sigma_i} \quad (7)$$

$$\frac{d_{32}}{D} = a(1 + b\phi)(We)^c \quad (8)$$

Calculation of the exponents of the experiments is presented in Eq. 9 as the suggested model.

$$\frac{d_{32}}{D} = 0.508(1 + 23.70\phi)(We)^{-0.6} \quad (9)$$

It describes the data with a correlation coefficient of 0.92. Fig. 5 shows that the measured experimental values lie within $\pm 7\%$ of those calculated using Eq. (9).

We compare both the suggested model and the Calderbank model in Table 4; error is calculated by the sum of least square method. The error of the Calderbank model is 10.91%, as opposed to 10% in the suggested model.

Evaluation separation of dispersion

Another moment that is evaluated and has industrial significance is the separation of phases in mixer-settler, and the aim of this section is acquiring the optimum

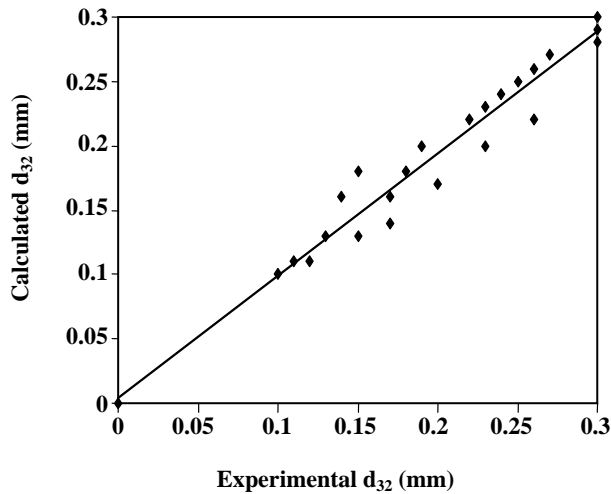


Fig. 5: Evaluation of experimental data with this model.

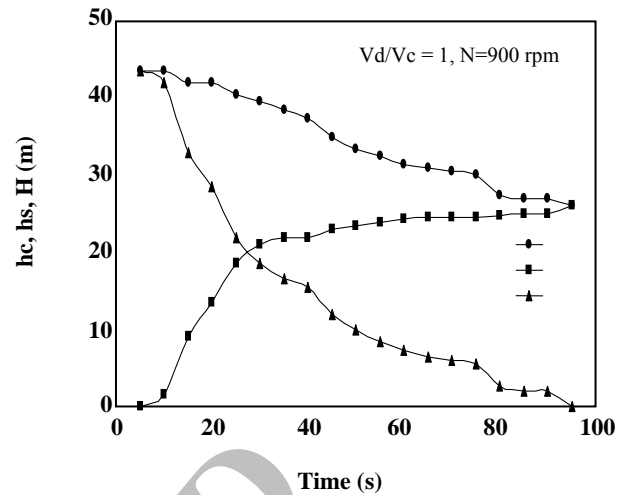


Fig. 7: Height of dispersion, Coalescent and separation band vs. time.

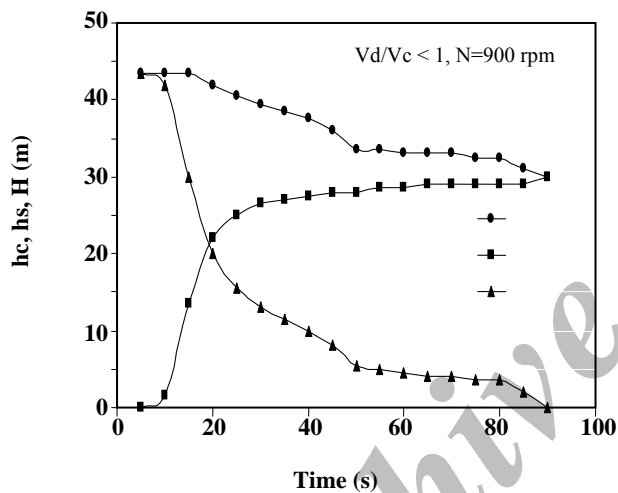


Fig. 6: Height of dispersion, Coalescent and separation band vs. time.

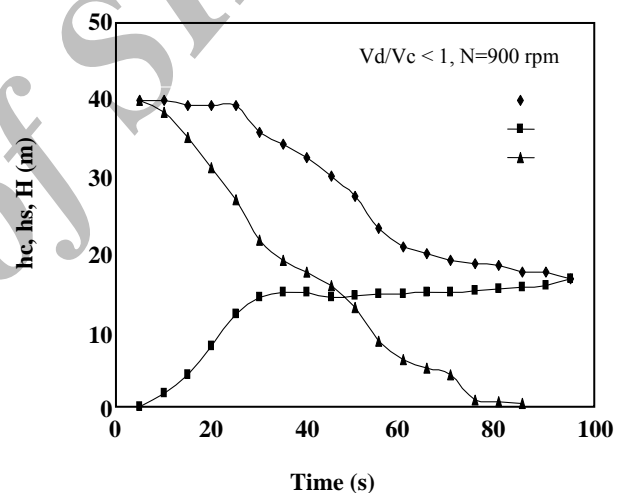


Fig. 8: Height of dispersion, Coalescent and separation band vs. time.

condition of impeller speed and volume fraction of organic phase to produce a large dispersion area for high extraction (Staszak & Prochaska 2006) [17].

This is illustrated versus time in Figs. 6, 7, 8, and 9 in different conditions as impeller speed and volume fraction of organic phase.

Coalition band (h_c), separation band (h_s), and height of dispersion (H) versus time in 900 rpm and different volume fractions of organic phase equal 0.3, 0.4, and 0.5, as shown in Figs. 6, 7, and 8. The relationship between h_c , h_s and H is presented in Eq. 10 (Ruiv *et al*, 2006 [12]; Chakra Borty *et al*, 2003 [2]).

$$H = h_c - h_s \quad (10)$$

Evaluation of the separation mechanism of dispersion is continued by studying h_c , h_s and H in constant volume fraction and different impeller speeds versus time in Figs. 7 and 9.

H_c , h_s , and H are gadded by photographing the settler at the time that dispersion enters the settler for 100 seconds and H equals zero, so two phases are separated, and all the tests at description section are done seven times, so they have high reliability.

Evaluation on separation mechanism of dispersion is scrutiny on both dispersion area and phases separation time; in other words, the larger dispersion wedge and surface area, the higher extraction efficiency. This wedge in several conditions of impeller speed and hold up quantity was studied in this article.

Table 6: Equilibrium data at 25°C.

ye (wt%)	xe (wt%)	ye (wt%)	xe (wt%)
0.00	0.00	7.66	6.13
0.26	0.21	7.75	6.20
0.98	0.78	10.00	8.00
1.41	1.13	10.88	8.70
1.93	1.54	11.88	9.50
2.98	2.38	13.50	10.80
4.23	3.38	14.00	11.20
6.66	5.33	15.00	12.00

Table 7: Experimental results at P =1bar and t =25°C.

N(rpm)	Vd(mL/min)	Vc(mL/min)	Xout	Yout	X*	Y*	Φ	D	E (%)	Kd(m/s)
750	59	45	10.71	4.29	3.43	13.39	0.50	1.10	52.4	1.84×10^{-5}
800	59	45	11.10	3.90	3.12	13.88	0.48	1.03	50.7	1.71×10^{-5}
900	59	45	10.02	4.98	3.99	12.52	0.46	1.25	55.5	2.08×10^{-5}
1000	59	45	9.56	5.44	4.35	11.95	0.40	1.36	57.5	2.26×10^{-5}
750	50	50	9.17	5.83	4.67	11.46	0.42	1.03	50.7	1.72×10^{-5}
800	50	50	9.57	5.43	4.34	11.96	0.41	0.95	48.5	1.58×10^{-5}
900	50	50	9.34	5.66	4.53	11.67	0.40	0.99	49.8	1.65×10^{-5}
1000	50	50	9.75	5.25	4.20	12.19	0.38	0.91	47.6	1.51×10^{-5}
750	42	53	9.45	5.55	4.44	11.81	0.36	0.97	49.2	1.62×10^{-5}
800	42	53	9.91	5.09	4.07	12.39	0.32	0.88	46.7	1.46×10^{-5}
900	42	53	10.02	4.98	3.99	12.52	0.28	1.09	52.0	1.81×10^{-5}
1000	42	53	8.82	6.18	4.94	11.03	0.26	1.11	52.6	1.85×10^{-5}

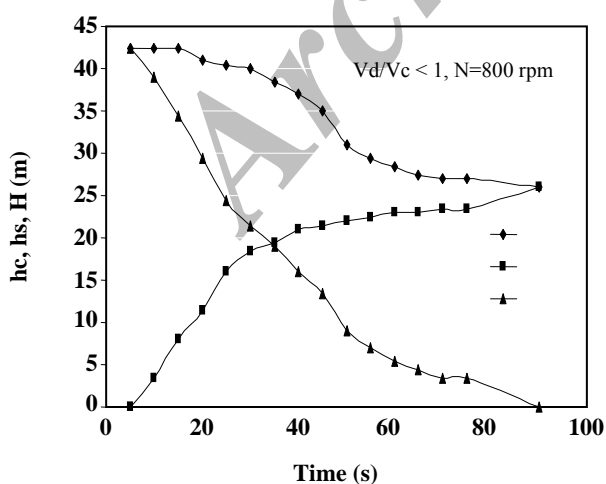


Fig. 9: Height of dispersion, Coalescent and separation band vs. time.

Evaluation of extraction efficiency, mass transfer coefficient in dispersed phase, distribution coefficient, and effect of different operational parameters

Information about equilibrium data was achieved by long residence time, and is given in Table 6.

The results of Evaluation of extraction efficiency, mass transfer coefficient in dispersed phase, distribution coefficient, and effect of different operational parameters are shown in Table 7.

The effect of impeller speed, hold up, and drop size on extraction efficiency at constant ratio of flow rates are shown in Fig. 10, and the effect of these factors on both mass transfer coefficient of dispersed phase and distribution coefficient are shown in Figs. 11 and 12, respectively.

Observation of Fig. 10, 11, and 12 indicates that the effect of impeller speed on extraction efficiency, mass transfer coefficient of dispersed phase, and distribution coefficient are similar, but in high impeller speed and $V_d/V_c < 1$, extraction efficiency reduces while the others increase.

Variation of efficiency versus impeller speed shows that increasing impeller speed causes an increase of extraction efficiency, inasmuch as increasing impeller speed makes the drop size reduce and settling time of droplets increase. The effect of hold up on extraction efficiency is similar to drop size, and we expect that when the hold up of dispersed phase increases, the extraction efficiency reduces. Moreover, hold up is impressionable with correct residence time of phases.

The effect of variation of Sauter drop size on extraction efficiency, mass transfer coefficient of dispersed phase and distribution coefficient is similar, except that $V_d/V_c < 1$ in the mass transfer mechanism converges because of aggregation of very tiny droplets, thereby increasing extraction efficiency and reducing both the mass transfer coefficient of the dispersed phase and the distribution coefficient.

The effect of holdup on extraction efficiency, mass transfer coefficient of dispersed phase, and distribution coefficient is similar to the Sauter drop size effect.

The effect of variation of hold up, Sauter drop size, and V_d/V_c on extraction efficiency, mass transfer coefficient of dispersed phase, and distribution coefficient is shown in Figs. 13, 14, and 15, respectively.

We observe that extraction efficiency, mass transfer coefficient of dispersed phase, and distribution coefficient under constant low and medium impeller speeds versus hold up, d_{32} , and V_d/V_c start reducing to minimum point then again resumes increasing.

At low and medium impeller speed with increasing in ratio of flow rates of phases, the hold up and Sauter drop sizes, extraction efficiency, mass transfer coefficient of dispersed phase, and distribution coefficient increase. However at high impeller speed and at the same conditions, these parameters reduce, then start to increase, again reduce to a minimum point, then resume increasing.

Variation of extraction with flow rate of dispersed phase in constant impeller speed expresses that with increasing of flow rate of dispersed phase or hold up, quantity of droplets grows. It means that percentage of dispersed phase is larger than continuous one. As a result, the mass transfer coefficient and extraction efficiency increase.

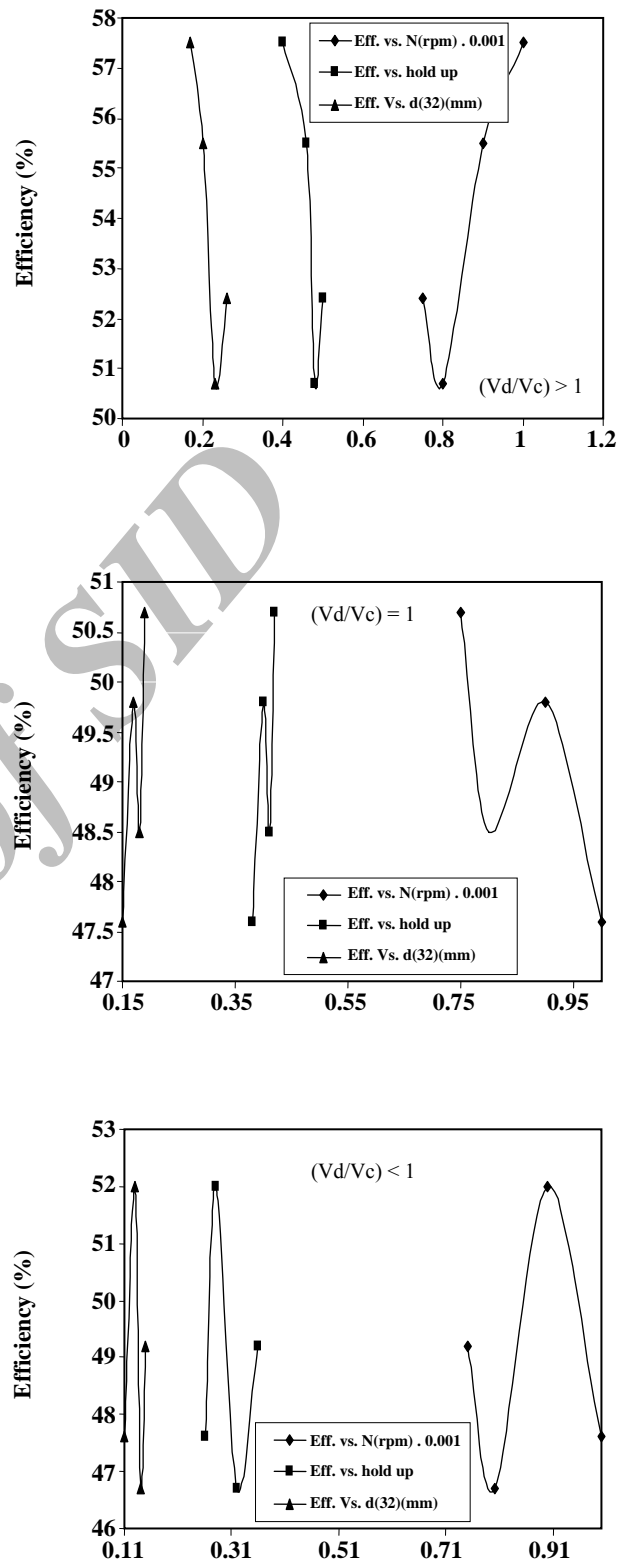


Fig. 10: Extraction efficiency vs. Impeller speed, hold up and drop size at constant ratio of Flow rates.

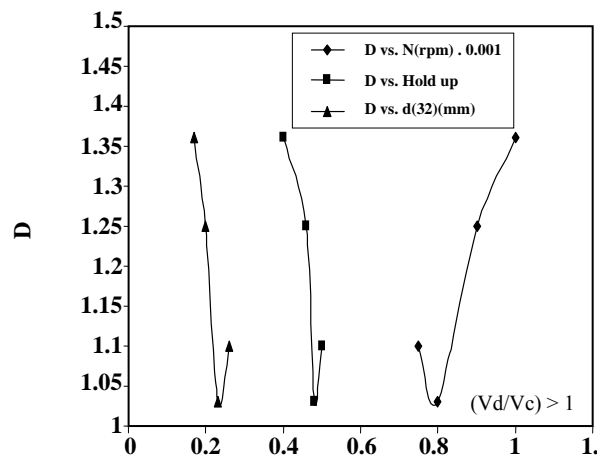
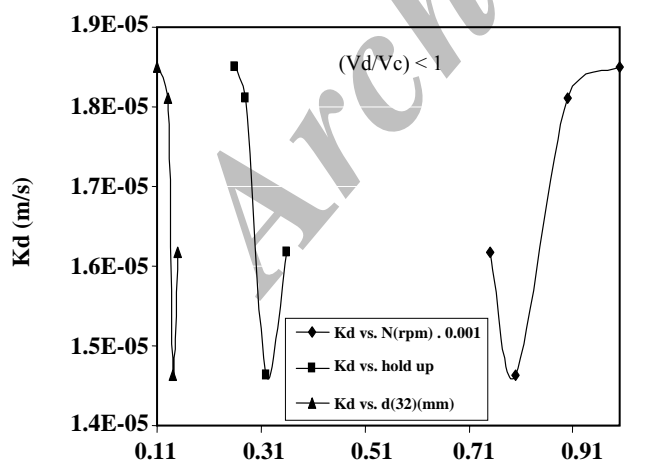
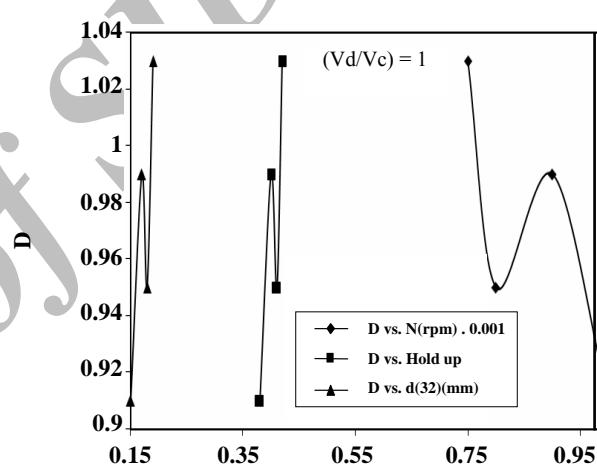
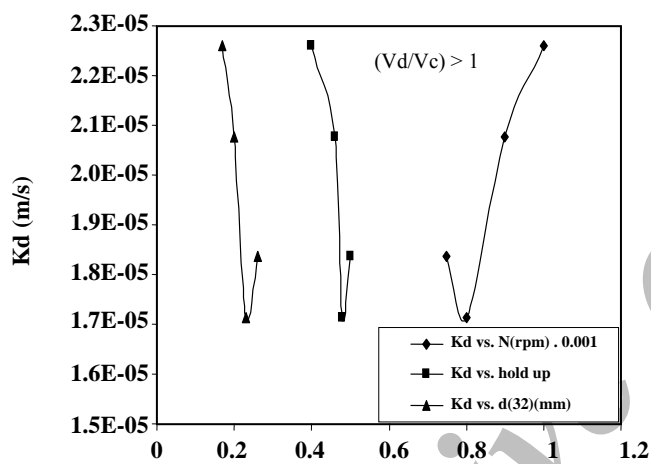
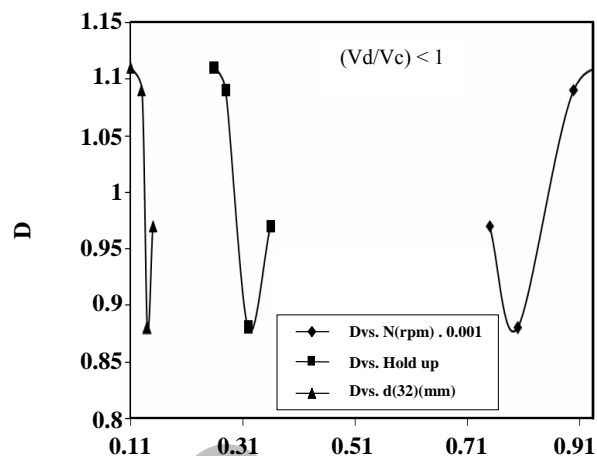
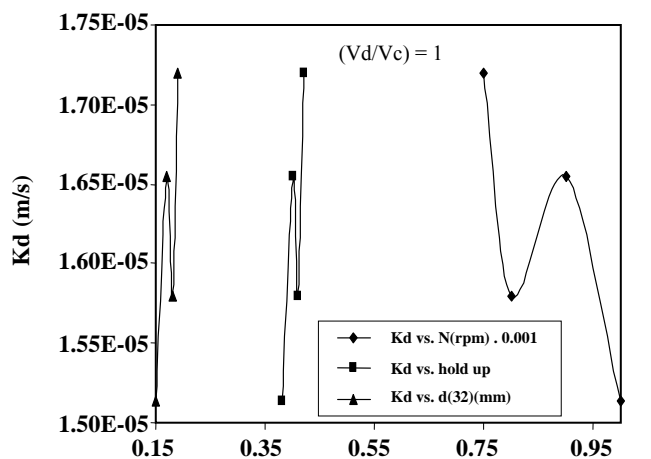


Fig. 11: Mass transfer coefficient of dispersed phase vs. Impeller speed, hold up and drop size at constant ratio of flow rates.

Fig. 12: Distribution Coefficient of dispersed phase vs. Impeller speed, hold up and drop size at constant ratio of flow rates.

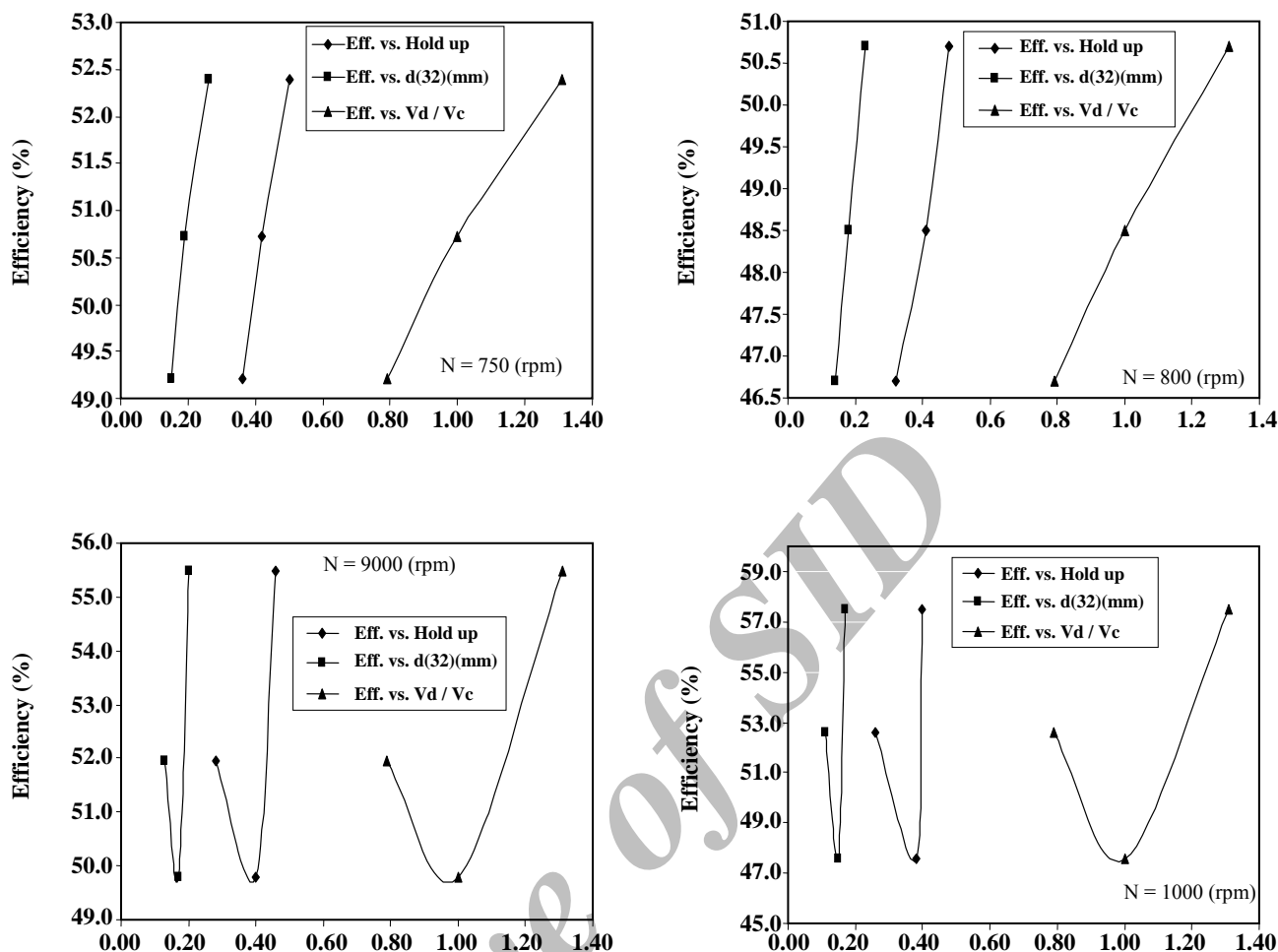


Fig. 13: Extraction efficiency vs. hold up, drop size and constant ratio of flow rates at constant impeller speed.

- When the rotor speeds were varied at a fixed dispersed phase throughput, the wedge length increased approximately 10% for every 100 rpm increment. At high energy input levels, that is, at about 1000 rpm the small drops (0.1-0.25 mm diameter) that were produced required a long time for coalescence.

- In constant ratio of flow rates, effect of impeller speed, drop size and hold up on extraction efficiency at $V_d/V_c < 1$ $V_d/V_c = 1$ is similar, but the direction of the curve inverts at high holdup.

- As for drop sizes and hold up, mentioned results are observed, inasmuch as inordinate aggregation of droplets occurred and although interface area increases, solute transfer from aqueous phase to organic phase reduces, so the mass transfer mechanism changes.

CONCLUSIONS

- The results obtained indicate that the behavior of a two-phase system during separation depends on the volume fraction of phases, intensity of agitation and performance of the mass transfer process.

- In other systems that are similar to our proposed system, the mean drop size varies with the rotor speed, dispersed phase holdup, and system physical properties. At low rotor speeds, the variation in drop size is greater than at higher speeds. These results are due to a drop residence time distribution in the mixer, and only a proportion of the drops spent a sufficient time in the discharge region of the impeller to cause breakup. No attempt was made to measure any variation in drop size with position in the vessel since, provided the critical

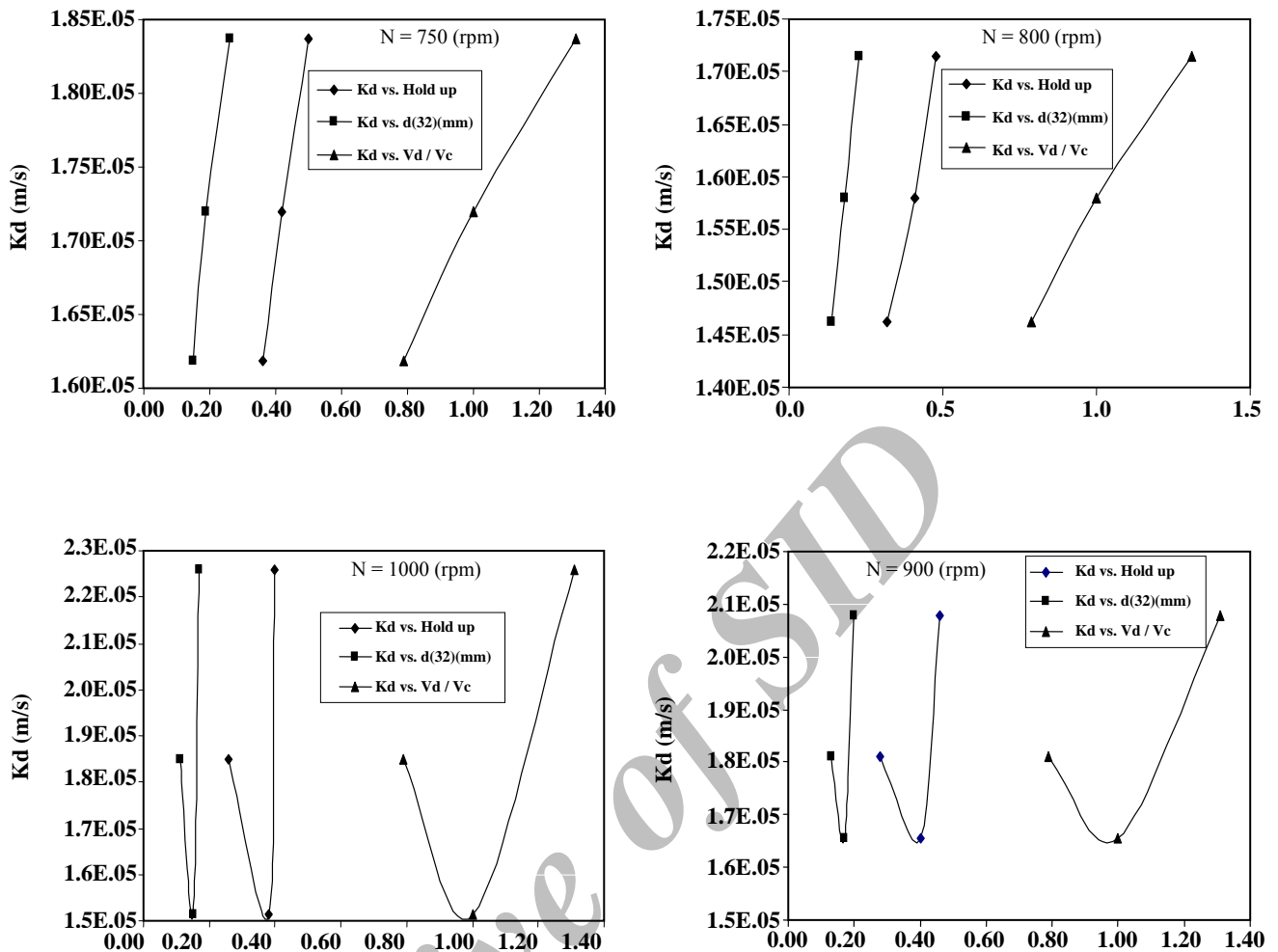


Fig. 14: Mass transfer coefficient vs. hold up, drop size and constant ratio of flow rates at constant impeller speed.

speed was exceeded, conditions in the relatively small vessel were homogeneous, as confirmed by the absence of any holdup profile. The Sauter mean drop diameter at constant rotor speed increases with holdup due to coalescence effects. As holdup increases, the probability of droplet collision followed by subsequent coalescence also increases.

- In general, an increase in throughput causes the axial velocity component of the drops to be much higher than the vertical component, thus hindering drop-interface coalescence and resulting in an increase in wedge length.

- It was observed that, after interrupting of dispersion in the settler, its shape is similar to the wedge, and after about one hundred seconds, it separated. With constant impeller speed, the height of dispersion increases directly against volume fraction, and both coalition and separation

bands decrease versus volume fraction, under these conditions, interface height decreases as well. In the constant volume fraction of phases, dispersion height increases directly versus impeller speed and both coalition and separation bands decrease versus impeller speed with interface height also decreasing.

- At constant impeller speed, with increasing ratios of phase flow rates or increasing solvent quantity, extraction efficiency, mass transfer coefficient of dispersed phase, and distribution coefficient also increase; however, at high impeller speeds, drop sizes are very small and the population is large, so the mechanism of mass transfer changes, then extraction efficiency, mass transfer coefficient of dispersed phase, and distribution coefficient start reducing to a minimum point then resume increasing.

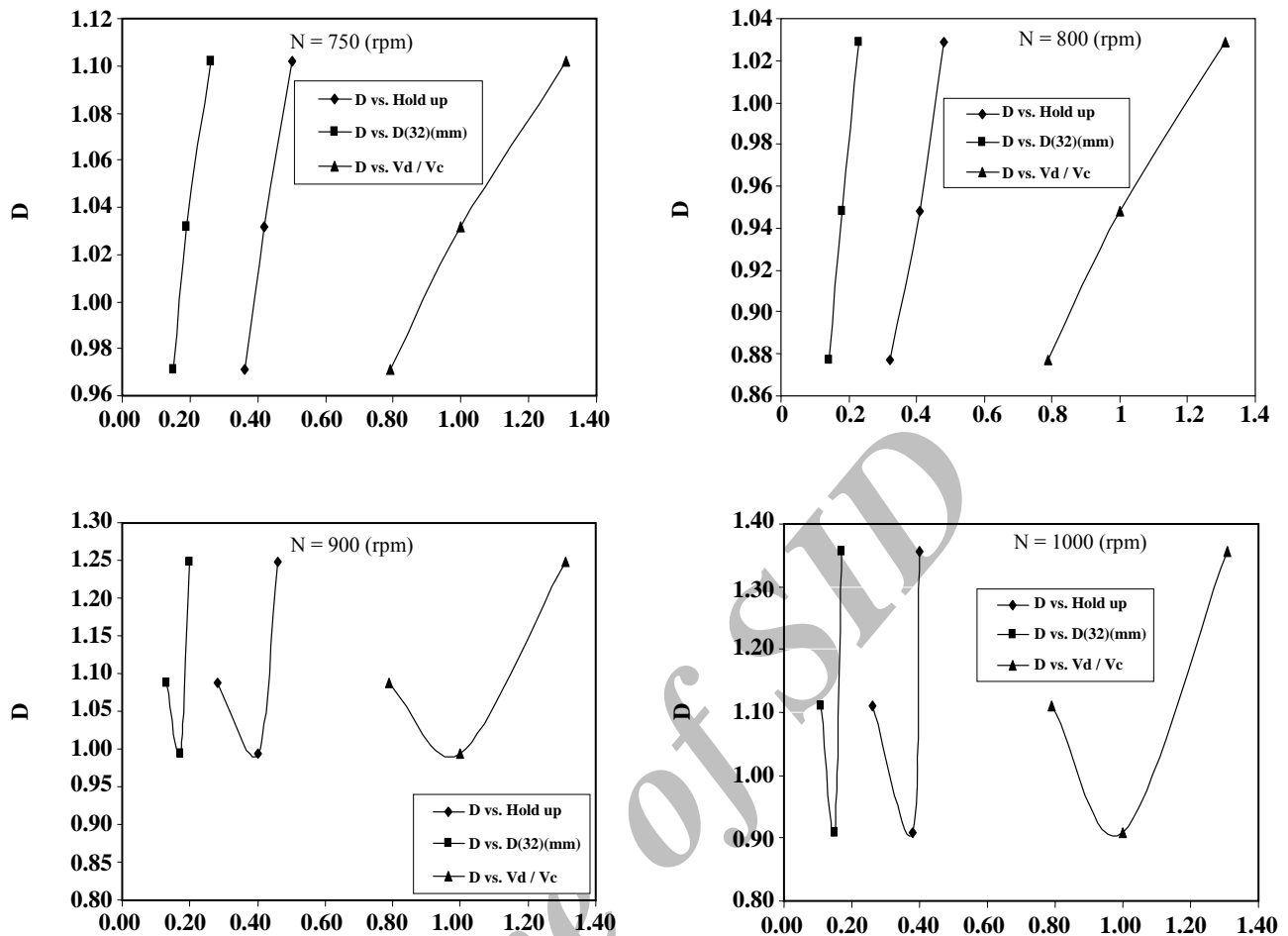


Fig. 15: Distribution Coefficients vs. hold up, drop size and constant ratio of flow rates at constant impeller speed.

- The more impeller speed makes, the more extraction efficiency; in other words, when it is increased, the droplet sizes decrease and their number increase; both surface area and mass transfer rate increase; these means either operational curve is further than equilibrium curve in the same condition or the set is in good operational condition; furthermore, in a special impeller speed and flow rate ratio of phases this procedure because of either inordinate droplet accumulation or changing in mass transfer mechanism makes a large decreasing in mass transfer coefficient.

- The more increasing in flow rate ratio of phases and impeller speed, the much hold up quantity; hence, dispersion phase hold up effects on extraction efficiency inversely and increasing in residence time of phases effects on directly.

Nomenclature

D	Mixing vessel diameter, mm
d	Drop diameter, mm
d_n	Average diameter of a group
d_{32}	Sauter mean drop diameter, mm
N	Rotor speed, rpm
n	Number of drops
V_d, V_c	Volumetric flow rate of dispersed and continuous phase, respectively, mL/min
We	Weber number, $We = \frac{D^3 N^2 d_{32}}{\sigma_i}$
\dot{m}_d	Mass flow rate of dispersed phase
y_{in}	Zirconium mass component in dispersed phase in inlet of mixer
y_{out}	Zirconium mass component in dispersed phase in outlet of settler

a	Particular surface area, m^2/m^3
a, b, c	Exponents in Eq. (2)
μ	Viscosity, cp
ρ_c	Continuous phase density, g/cm^3
σ	Interfacial tension, Dyne/cm
ϕ	Dispersion phase hold up
H	Height of dispersion, mm
hc	Coalition band, mm
hs	Separation band, mm
N	Mass transfer flux, kg/m.s
Kd	Overall mass transfer coefficient in dispersed phase, m/s
Cd,Out	Solute concentration in dispersed phase in outlet of settler, kg/m^3
C^*d	Solute equilibrium concentration in dispersed phase, kg/m^3
$k=Vd/Vc$	Ratio of volumetric flow rate of dispersed to continuous phase
M	Concentration of solute, g/L

Received : Dec. 30, 2009 ; Accepted : Mar. 15, 2010

REFERENCES

- [1] Ali A.M.I., Ahmad I.M., Daoud J.A., CYANEX 272 for the Extraction and Recovery of Zinc from Aqueous Waste Solution Using a Mixer-Settler Unit, *Sep. Purif. Technol.*, **47**, p. 135 (2006).
- [2] Chakra Borty M., Bhattacharya C., Datt S., Effect of Drop Size Distribution on Mass Transfer Analysis of the Extraction of Nickel (II) by Emulsion Liquid Membrane, *Physicochem. Eng. Aspects.*, **224**, p. 65 (2003).
- [3] Chouai, M. C., Le Lann M.V., Gourdon C., Casamatta G., Use of Neural Networks for Liquid-Liquid Extraction Column Modeling: an Experimental Study, *Chem. Eng. Process.*, **39**, p. 171 (2000).
- [4] Dehghani T.R.F., Foster N.R., Current Issues Relating to Anti-Solvent Micronisation Techniques and Their Extension to Industrial Scales, *J. Supercrit. Fluid.*, **21**, p. 159 (2001).
- [5] Dehkordi M., Application of a Novel-Opposed-Jets Contacting Device in Liquid-Liquid Extraction, *Chem. Eng. Process.*, **41**, p. 251 (2002).
- [6] Giraldo-Zuniga D.A., Jane Coimbra S.R., Luis Minim A., Edwin Garcia Rojas E., Axial Mixing in a Graesser Liquid-Liquid Contactor Using Aqueous Two-Phase Systems, *Chem. Eng. Process.*, **44**, p. 441 (2005).
- [7] Giraldo-Zuniga, D., Jane Coimbra S.R., Luis Minim A., Edwin Garcia Rojas E., Current Issues Relating to Anti-Solvent Micronisation Techniques and Their Extension to Industrial Scales, *J. Super. Crit. Fluid.*, **21**, p. 159 (2001).
- [8] Giraldo-Zuniga D.A., Jane Coimbra S.R., Luis Minim A., Edwin Garcia Rojas E., Dispersed Phase Hold-Up in a Graesser Raining Bucket Contactor Using aqueous Two-Phase Systems, *J. Food Eng.*, **72**, p. 302 (2006).
- [9] Gottlieb K., Grinbaum B., Chen D., Stevens G.W., The Use of Pulsed Perforated Plate Extraction Column for Recovery of Sulphuric Acid from Copper Tank House Electrolyte Bleeds, *Hydrometallurgy.*, **58**, p. 203 (2000).
- [10] Pinto, G. A., Durañob F.O., Fiu'z A.M.A., Guimaraes M.B.L., Novais Madureirac C.M., Design Optimisation Study of Solvent Extraction: Chemical Reaction, Mass Transfer and Mixer-Settler Hydrodynamics, *Hydrometallurgy.*, **74**, p. 131 (2004).
- [11] Rajmane, M.M., Sargar B.M., Mahamuni S.V., Anuse M.A., Solvent Extraction Separation of Zirconium(IV) from Succinate Media with *N-n*-Octylaniline, *J. Serb. Chem. Soc.*, **71**, p. 223 (2006).
- [12] Ruivo R., Paiva A., Simões P.C., Hydrodynamics and Mass Transfer of a Static Mixer at High Pressure Conditions, *Chem. Eng. Process.*, **45**, p. 224 (2006).
- [13] Ruiz M.C., Lermenda P., Padill R., Drop Size Distribution in a Batch Mixer under Breakage Conditions, *Hydrometallurgy*, **63**, p. 65 (2002).
- [14] Saien, J., Ebrahimzadeh Zonouzian S.A., Molaei Dehkordi A., Investigation of a Two Impinging-Jets Contacting Device for Liquid-Liquid Extraction Processes, *Chem. Eng. Sci.*, **61**, p. 3942 (2006).
- [15] Schloman Jr.W.W. Processing Guayule for Latex and Bulk Rubber, *Ind. Crop. Prod.*, **22**, p. 41 (2005).
- [16] Schmidt S.A., Simon M., Attarakih M.M., Lagar G. L., Bart H., Droplet Population Balance Modeling-Hydrodynamics and Mass Transfer, *Chem. Eng. Sci.*, **61**, p. 246 (2006).
- [17] Staszak K., Prochaska K., Adsorption at the Liquid-Liquid Interface in Mixed Systems with Hydrophobic Extractants and Modifiers 2. Dynamic Interfacial Tension at the Hydrocarbon/Water Interface in Binary Mixed Systems, *J. Colloid. Interf. Sci.*, **294**, p. 411 (2006).