Hydrodynamic behavior and mass transfer coefficients of single liquid drop in extraction column

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Hydrodynamic behavior and mass transfer of several liquid-liquid system has been measured in 150.0 mm column diameter. The column was operated in tow different modes as a spray tower and a perforated plate Experiments were performed using different tower. chemical systems and different needles to obtain drops of different diameters. The relationship between the terminal velocity (V_t) and the down flow of the continuous phase was examined experimentally. Mass transfer coefficients (MTC_s) have been measured for single liquid drops rising through non flowing and flowing water and have been compared with the coefficients measured for free rise conditions in an empty column for different systems. The MTC_s were determined as a function of drop size, mass transfer direction and down flow of the continuous phase. From the data obtained, it is concluded that the drop size is key variable affecting hydrodynamics and mass transfer processes. The relative velocities between drops and the down-flowing continuous phase were found to decrease as a down flow rate increased. The effect of continuous phase velocity on The MTC is relatively significant with a slight increase in drop size occurring with increasing flow rate. The degree of enhancement of the dispersed phase film coefficients for mass transfer due to the presence of plates has been ascertained. Finally the coefficients for mass transfer from the dispersed phase into the continuous

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phase (d \rightarrow c) are slightly higher than those for the opposite direction, (c \rightarrow d).

Keywords: Extraction columns, Hydrodynamic behavior, single liquid drop, mass transfer.

1. Introduction :

In the field of solvent extraction processes various types of equipment have been developed during the last few decades. Therefore the user is faced with the critical choice between contactors and most of the time has to decide according to subjective criteria. Nowadays various extractors which are operated with and without a supply of mechanical energy are available in industry spray and perforated-palate (sieve plate) columns are commonly used in commercial liquid -liquid extraction systems. In these towers many drops are formed simultaneously and rise (or fall) in a swarm. Spray columns are the simplest of liquid -liquid extractors for contacting the continuous phase with counter current stream of the dispersed phase droplets. Spray column have been little used in industry despite their simplicity, high though put and low cost. This is because the lack of internals results in a strong circulatory flow of continuous phase in the same direction as the droplets, with a return flow around the column wall (1). Consequently, these column approach the fully back mixed conditions with a performance equivalent to only1-2 stages. Hence, it is used only when the number of overall



transfer units involved in the separation process is relatively small.

One of the ways to increase the mass transfer rates between the dispersed and the continuous phase is to provide for frequent coalescence and redispersion of the drops especially for the systems which show high internal circulation in the drop during formation at orifice or nozzles (2). Perforated plate extraction towers are unique in the they provide repeated coalescence and redispersion for the drops and offer the advantage of cross flow of the continuous phase in addition to yielding high extraction coefficients with practically no axial mixing. The perforated plate columns may be considered as a series of short spray columns arranged one above the other. In practical, however, thee are numerous application of liquid-liquid extraction where perforated columns are preferred (3).

2. Scope of present work:

The improvement of the design method of any type of column depends on the understanding of drop size, drop velocities, residence time distribution and mass transfer coefficients. The rate of mass transfer is affected by the rate of the formation of the droplets, their rate of passage through the continuous phase and finally their rate of coalescence. Mass transfer to and from drops moving in a continuous phase is common to many industrial processes, especially those involving liquid-liquid extraction and liquid phase reaction. The information obtained from simple



single drop experiments supply the fundamental data required for modeling and may be used to predict the behavior of extraction columns. Observation of liquid-liquid extraction form single drops has advantage that the extraction can be carried out under known conditions of interfacial area and time of contact. The results should be directly applicable to spray towers and perforated-plate extractors and should help to explain the mechanism of mass transfer in other types of equipment.

In the previous paper (4), we presented experimental results on mass transfer rate for the following systems: toluene – acetone – water, toluene – acetic acid – water and kerosene – benzoic acid – water. The present work aims at the prediction of mass transfer rates from single drop to a continuous phase and investigate the effect of drop size, mass transfer direction, height of compartment and continuous phase velocity on the mass transfer coefficients of single liquid drop in a perforated plate (sieve-plate) and spray columns using different systems.

Since, knowledge of particle velocity is of fundamental importance for the description of mass transfer process, in order to estimate mass transfer to or from droplets, it is necessary to consider some aspects of the fluid mechanics of a single particle in free motion through a continuous fluid phase. Aspects which are directly related to mass transfer are the particle velocity relative to the continuous phase (5). Hence, the relevant hydrodynamics to mass transfer consideration will first be reviewed briefly, with



drop size and terminal velocity because the primary focus of this paper on mass transfer of single liquid drop.

3.Systems :

The systems investigated in this work were, toluene/ water (system 1) and kerosene / water (system 2) for hydrodynamic studies, toluene / phenol/water (system 1) and toluene/ benzoic acid / water (system 2) for mass transfer studies.

The physical properties of the systems used are given in tables 1, 2. The dispersed phase is specified first, followed by the solute and the continuous phase. The main difference between the liquid-liquid systems which were selected was interfacial tension which was regards as the primary characteristic of each system (6). Phenol and benzoic acid were selected as the solutes in this work since it is non-corrosive, permitting common materials of construction to be used; it also has the advantage the analysis of the column sample is comparatively simple. Solute concentration were determined by measurement of absorbency relative of ultraviolet where rav а spectrophotometer (UV160 Shimadzu) was used for measurement of relative absorbency of the sample placed in 2 mm size cell. Calibration charts for relative absorbency against concentration were prepared by measuring the absorbency of solutions of known relative solute concentration. The organic liquid were special- grad chemicals and were used without concentration of each



solute, used in this work was 1000 ppm with transfer from aqueous to organic.

Table 1 : Physical properties of the systems used(hydrodynamic studies)

System 1: toluene / water						
p _c	p_{d}	μ_{c}	μ_{d}	γ		
(kg/m ³)	(kg/m ³)	(kg/ms.10 ³	(kg/ms.10 ³	(mN/m)		
))			
998	862	0.98	0.59	34		
System 2: kerosene / water						
988	807	1.1	1.47	42		

Table 2: Physical properties of the systems studied(mass transfer studies).

System 1: toluene / phenol / water						
p _c (kg/m ³)	p _d (kg/m ³)	μ _c (kg/ms.10 ³)	μ_{d} (kg/ms.10 ³)	γ (mN/m)		
998	876	1.01	0.61	43.5		
System 2: toluene / benzoic acid / water						
999	796	1.08	1.65	38.5		

3.1 Single drop apparatus:

The experimental apparatus were essentially the same as described in the previous paper by the authors .

A summary of column details is given in table 3. A schematic diagram of the apparatus is shown in fig.1, Main parts of the apparatus consisted of Perspex glass extraction column 0.150m diameter, a glass funnel for sample



collection and one of several sizes of glass capillary for drop formation.

The rising time of the drops through given distance was measured with a stop watch. Solvents of different types were pumped through a needle using a syringe pump (Razel scientific instrument, type A-99). The equivalent spherical drop diameter, d, was calculated knowing the flow rate and



Fig.1. Schematic diagram of experimental apparatus

counting the number of drops formed in unit time. Drop were generally spherical, moving without oscillation. All the experiments were conducted with the aqueous phase continuous and the organic phase dispersed at laboratory temperature $25 \pm 2^{\circ}$ C.



Column diameter, mm	150
Plate hole diameter, mm	5
Number of holes	37
Plates thickness mm	3
Plate spacing, variables, mm	20,40.60
Area of perforations (%)	42

Table 3 : Perforated column details

4. Experimental Procedure

Prior to the commencement of measurement the apparatus was thoroughly cleaned and the system run to achieve saturation of the phases (for the mass transfer experiments). The column was filled with the aqueous phase (distilled water) before the solvent (dispersed phase) is fed. Rotameters on the lines to and from the column enabled accurate flow settings and adjustment to be made thus ensuring that balanced flows were rapidly achieved and then maintained.

A positive displacement pump was used to introduce the organic phase into the bottom of the column. When the dispersed phase reaches the top section of the glass needle the pump flow rate setting was reduced so it almost equals the atmospheric pressure ensuring the dispersed phase level inside the glass needle remains constant. The syringe pump was kept running while the column was filled with distilled water(7). The pump and syringe were mounted at a higher level in order to avoid back flow of solvent in the syringe. After each experiment the column was thoroughly cleaned with decontaminating agent

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(Decon 90) to prevent the problem of contamination. Experiments were performed using different glass needles to obtain drop of different size. The equivalent spherical drop diameter was calculated, knowing the flow rate from the svringe pump and counting the number of drops formed and measuring the time if formation, using an electronic stop watch. At least 300 drops were timed for each run, after a period that is enough to reach the steady state conditions (8). Drops were spaced more than 30 mm apart typically, sufficient to avoid interactions according to skelland (9). In the perforated column, the droplet leaving the plates were allowed to rise a measured distance through the aqueous phase before being collected by suction through a small inverted funnel. The interfacial area in the funnel was kept small by occasionally pulling the drops into a pipette.

5.Inerpretation of data:

The overall mass transfer coefficients for drops of known size may be measure by passing solvent drops of known composition through an aqueous phase of known composition and collecting drops in an inverted glass funnel at the top of the column (10). Collection of solvent drops in a glass funnel, covering about a quarter of the cross-section area of the column, was found satisfactory. The funnel was immersed to an extent such that the interface where drops coalesced was in the funnel stem, (11).



Analysis of solvent drops after passage through the column in a known time allows an overall time-averaged mass transfer coefficient to be calculated from the following equation:

$$V dc / dt = k_{od} A_d (C - C^*).$$
 (1)
Integrating eq. (1) yields.
$$(C_o - C_i) / (C^* - C_i) = (1 - \exp(-6 k_{od} t / d)),$$
 (2)
$$K_{od} = - (d / 6t) \ln(1 - E),$$
 (3)
ere:

Where:

E = ($C_{i}-C_{o}$) / ($C_{i}-C^{\ast}$) and C_{i} is the inlet and C_{o}

outlet solvent concentration, and since there was no solute initially in the water, the drop concentration in equilibrium with the aqueous phase, $C^* = 0$. Drop formation time at the needle was easily found by counting drops at a fixed flow rate over a given time. This drop formation was included in the time used for calculating drop diameter (d).

6. Results and discussion:

6.1. Hydrodynamic terminal velocities of single drops

Study of the formation and character of movement of drops is an important for understanding the extraction process. The terminal velocity of a liquid drop in liquid medium is the free fall or rise, depending on the relative density, velocity of a single isolated drop in the gravitational field. Experimental values of terminal velocities of single drop falling or rising in



liquid –liquid systems have been correlated by Hu and kintener (12), klee and treyball (13), Johnson and Braida (14), and Grace (15). The latter utilized data from several sources and conducted many experiments of their own, enabling their correlation to cover a wide range of variables.

The experimental terminal velocities of our systems are plotted versus drop diameter in fig 2. Also shown are the terminal velocities for these drop rising singly as predicted by the correlation of Grace for comparison with measured values. The terminal velocity is observed to increase with increasing droplet diameter. The reasonable agreement indicates that all drops in this work experienced some internal circulation.

6.2. Effect of Continuous phase velocity

Fig. 3 shows the profile of down flow of the continuous phase against the terminal velocity under various flow rates conditions. The terminal velocity decreases as the down flow rate increases, this due to the increase on the residence time of the drop inside the column. On account of this drag and skin friction increase which apparently cause a decrease in the terminal velocity. This suggested that drops were taking a longer path through the column dependent on the down flow. Since liquid were initially circulate over the top cross sectional area of the column using a liquid



distributor, this circulation has a significant effect on increasing the rising time of drop and hence decreasing the terminal velocity.





6.3. Mass transfer MTC in spray and perforated columns.

Fig. 4 shows the mass transfer coefficient for spray and perforated columns for system 1, 2. The mass transfer coefficient in the presence of a sieve plate is higher



than that without plate. The obtained results are in agreement with observation of other qualitative investigator (16, 17, 18). This is due to the fact that during the operation of a spray columns the continuous phase is relatively turbulent through the column leading to axial mixing in that phase. This reduces the extraction rate because of the deviation from true counter current behavior. In a sieve plate contactor the distortion of the drop by direct contact with the edge of plate holes during the passage disturbs the the concentration profile inside the droplet and increases the mass transfer coefficient. The enhancement of up to about 80% is presumed under the influence of plates, are far greater than the values of 30% obtained in the absence of plate due to the disturbing action of plates on drops.



Fig.4. Mass transfer coefficient for system 1.2.



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6.4. Effect of drop size and direction of solute transfer

Fig. 5 shows the dependence of the average mass transfer coefficient on drop size and direction of the mass transfer for system 2. The coefficients of mass transfer from the continuous to the dispersed appears $(c \rightarrow d)$ to be rather smaller than those obtained for the opposite direction of transfer (d \rightarrow c). The difference in the coefficient is, however, small. With slightly increase in drop size from $(d \rightarrow c)$. It is significant, however, that the result follows the same trends as those for the opposite direction of transfer. Direct comparison of the results is difficult owing to the variation of drop size with direction of solute transfer. With solute transfer from $(d \rightarrow c)$ the drop size increased. This effect can be attributed to the increase in coalescence tendency of the drops promoted by the direction of solute direction. Values for the larger drops are bounded by the Handlos- Baron and the Hadamard relation for circulating drops. In addition the mechanism of mass transfer is known to be dependent on drop size, with the mass transfer mechanism varying from , molecular diffusion for very small drops, which behave as a rigid spheres, to the development of internal droplet circulation, oscillation and turbulent eddy transfer mechanisms as the drop size is increased. Drop size produced were always less than plate hole size, drop would hit plates, move sideways, then move upward to



the next plate, only occasionally did drops remain at a plate.



Fig. 5. Dependency of the MTC on drop size and direction of solute transfer for system 2.

It is well known that surface instability or Marngoni effect can increase solute transfer rates appreciably (19, 20). This instability is also known to affect the coalescence tendency of the liquid drops, depending on the sign of the gradient of the variation in interfacial tension with solute concentration and the direction of solute transfer.

6.5. Effect of Continuous phase velocity

A comparison of the mass transfer results obtained with the freely rising drops, under conditions of zero continuous phase velocity and those obtained at different velocities of the continuous phase for spray and perforated plate column. Thus provides an



interesting measure of the effect of the continuous phase on the mass transfer process different operating conditions.

The effect of the continuous phase velocity on the mass transfer coefficient was found to be significant (2) with only a slight increase in the drop size occurring with increasing the continuous phase velocity. The enhancement factor, E, defined as the ratio of mass transfer coefficient at zero down flow to that at a given value of down flow, k_{od}/K_{odo} , as a function of continuous phase velocity (figs 6, 7) show how increase the continuous phase velocity increases the ratio. The study of the continuous phase velocity was also useful in that it enable a comparison between the unsteady state (22) and the stationary phase methods for single liquid drops (23). The obtained results were in good agreement with those of other workers (24-26). The effect of down flow on the dispersed phase mass transfer coefficient is associated directly with movement of drops through holes. Other important effect are obtained by reducing drop relative velocities (27) and increasing in contact time leading to reduction in k_d and a change in the product $k_d t / d$ which affect the benefit fraction extraction. The main is the enhancement of mass transfer though the droplet/ continuous phase interface as demonstrated by other investigators (28-30).



6.6. Effect of compartment height

The mass transfer coefficients slightly increased by increasing the height through which drops raise after leaving the plate (fig 8, 9). This indicated that much of the mass transfer occurred at or near perforation at which the drops were formed. For liquid- liquid extraction, it can be concluded that efficiencies in the order of 70% to 80% are normally obtained for a perforated plate. The degree of enhancement of the dispersed phase film coefficient due to the presence of plates has been ascertainec. The experiments using variable rise heights indicate that most of the mass transfer occurs within a few centimeters of the plate. These considerations might suggest that a multi-plate column with high efficiency could be designed with a very close plate spacing. A common plate design with a free area of 42% was used throughout the experiments.



Fig. 6. Effect of continuous phase velocity on the enhancement factor for phenol / toluene / water.





Fig. 7. Enhancement factor vs. Vo for benzoic acid/ toluene / water in perforated plate column.

In order to minimize the effect of axial mixing on column mass transfer it would therefore appear preferable to limit industrial plate spacing to between 40 and 80mm {31}. Experience has shown that plates spacing between 40 and 100mm are optimal larger plate spacing favor backmixing and hence reduce the efficiency of the column. On the other hand, smaller plated spacing lead to a strong decrease of the maximum throughput (32). However, care must be taken to control the formation of very fine drops which would lead to entertainment problem in counter-current operation (33-35).





Fig. 8. Effect of compartment height on mass transfer coefficients for benzoic acid/toluene/ water.



Fig. 9. Effect of compartment height on mass transfer coefficients for benzoic acid/toluene/ water.

7. Conclusion:

The drop size is key variable affecting hydrodynamics and mass transfer processes the relative velocities between drops and the down-flowing continuous phase were found to decrease as a down flow rate increased. The mass transfer coefficient in the presence of plates is higher than that without plates. The direction of solute transfer has been shown to affect the mass transfer coefficient to a very



significant effect. The coefficients for mass transfer from the dispersed phase into the continuous phase direction (d c) are slightly higher than that for the opposite direction of transfer(c d). The mass transfer coefficient were found to be enhanced by circulation of the continuous phase. The effect of continuous phase velocity on the mass transfer coefficient is relatively significant with a slight increase in the drop size occurring with increasing flow rate. The mass transfer coefficient slightly increased by increasing the height through which drops raise after leaving the plate.

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Notations :

d Drop diameter m,

c→d Continuous to dispersed phase solute direction,

d→c Dispersed to continuous phase solute direction,

E Degree of extraction, $(C_i - C_o)/(C_i - C^*)$,

 $K_{\rm c}$, $k_{\rm d}$ Contin. And disp. Phase mass, transfer coefficient, m/s

k_{od} Overall mass transfer coefficient, m/s.

 t_f , t_r Drop formation time, drop rise time respectively, s. V_c , V_d Continuous phase velocity, dispersed phase velocity, m/s.



Greek Letter

 μ Viscosity kg/m s.

- p Density kg/m3, and
- γ Interfacial tension, mN/m

Subscripts

c Continuous, andd DispersedReferences



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السلوك الهيدروديناميكي ومعامل انتقال ألكتله للقطرة الوحيدة ألسائله في أبراج الاستخلاص

د. هاني أحمد قاسم دماج ، أستاذ الهندسة الكيميائية المشارك - قسم الهندسة الكيميائية، كلية الهندسة والبتـرول جامعة حضرموت للعلوم والتكنولوجيا - الجمهورية اليمنية- المكلا

تم في هذا البحث قياس معامل انتقال الكتلة والسلوك الهيدروديناميكي لأنظمة عديدة من السوائل في عمود قطره ١٥٠ مم وتم تشغيل العمود في نظامين مختلفين كبرج انتشار وبرج ذو صوانى مثقبة . وتم إجراء التجارب باستخدام أنظمة كيميائية مختلفة وأناديب ذات أقطار مختلفة بهدف الحصول على قطرات ذات أحجام مختلفة . تم دراسة العلاقة بين سرعة القطرة وتأثير معدل السربان للطور السائل على السرعة وكذلك تم قياس معامل انتقال الكتلة للقطرة الوحيدة السائلة أثناء صعودها فج الماء الساكن والماء المتحرك ومقاربة النتائج بتلك في برج الانتشار وتم تحديد معامل انتقال الكتلة كداله لحجم القطرة واتجاه انتقال الكتلة الهيدروديناميكي وانتقال الكتلة وان السرعة النسبية للقطرة تنخفض كلما زاد السريان العكسى وإن تأثير سرعة الطور المستمر على معدل انتقال الكتلة وإضح نسبيا مع زيادة في حجم القطرة كلما زادت سرعة الطور المستمر. وإن درجة التعجيل في انتقال الكتلة نتيجة لوجود الصواني قد تحققت . وأخيرا فإن معاملات انتقال الكتلة من الطور المشتت إلى الطور المستمر أعلى منه في الاتجاه العكسى أى من المستمر إلى المشتت. الكلمات ألمفتاحيه :أبراج الاستخلاص، السلوك الهيدروديناميكي القطرة الوحيدة ألسائله ، معامل انتقال ألكتله.

