Effect of surfactants on mass transfer coefficients of single liquid drops

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The effect of surfactants on the rate of mass transfer from single liquid drop rising through a stationary and moving phase was analysed using a spray column 0.15 m diameter. The surfactants used are Carboxy Methyl Cellulose (CMC) and Sodium Dodecyle Sulphate (SDS). The system acetic acid/toluene/water was employed throughout this study with solute transfer direction from the continuous phase to the dispersed and vise versa. Variables investigated were concentration and type of surfactant, direction of solute transfer, drop size and flow rate of the continuous phase. It was observed that the surfactants decrease the mass transfer coefficients drastically compared to the case with the pure systems. From the data obtained, it is concluded that the concentration and type of surfactant have a pronounced effect on the rate of solute transfer as well as drag coefficient of the rising drops. The surfactants lengthen the life time of the droplet inside the column. The Mass Transfer Coefficient (MTC) was found to decrease with increasing the concentration of surfactants. It was found that the surfactant decrease MTC by an amount ranging from 20 to 50 % depending on type of surfactant added. On the other hand, the mass transfer coefficient was found to increase with increasing the velocity of the continuous phase for surfactant-free solutions.

تم في هدا البحث دراسة تأثير المواد المنشطة للسطح مثل صوديوم كربوكسى مثيل سليولوز CMC والصوديوم دوديسيل سلفات SDS على سلوك القطرات السائلة الوحيدة أثناء صعودها في عمود استخلاص قطرة ٠,١٥ م وذلك باستخدام النظام الكيميائي حمض الخليك والتلوين والماء و تم دراسة تأثير انتقال المذاب (حمض ا لخليك) من الطور المستمر(المائي) إلى الطور المشتت (العضوي) وبالعكس ولقد أجريت التجارب بهدف دراسة تأثير كل من تركيز ونوع المادة المضافة (SDS or CMC) وكذلك اتجاه انتقال المذاب وحجم القطرة ومعدل السريان للطور المستمر على معدل انتقال المادة وأثبتت التجارب أن إضافة هذة المواد تقلل من معدل انتقال المادة بالمقارنة بالنظام النقي الخالي من تلك المواد ومن خلال النتائج التي تم الحصول عليها أتضح أن تركيز ونوع المواد المضافة له تأثير واضح على معدل انتقال المادة أثناء صعود القطرة فى عمود الأستخلاًص وأتضح أن معامل انتقال المادة ينخفض بزيادة تركيز هده المواد بنسبة ٢٠-٥٠% تبعا إلى نوع المادة المضافة وظروف إجراء التجارب كذلك فإن معامل انتقال المادة يزداد بزيادة سرعة الطور المستمر في حالة المحلول الخالي من تلك المواد بالمقارنة بوجود تلك المواد.

Keywords: Single liquid drop, Mass transfer coefficients, Surfactants, Sodium dodecyle sulphate, Sodium carboy methyl cellulose

1. Introduction

The behaviour of liquid drops from nozzles has long been a topic of interest because of its occurrence in a wide variety of engineering applications, such as distillation, extraction processes, spraying and emulsifying technologies among others. Hence, for understanding the column performance and developing adequate column design procedure, the hydrodynamic behaviour and mass transfer of single drops should be considered. The information obtained from simple single drop experiments supplies the basic data required for modelling, design and may be used to predict the behaviour of extraction columns. The effect of impurities or surfactants on the fluid dynamic or mass transfer may be taken into account by drop experiment carried out with the original substance to be used in the process [1].

Impurities, which may be surface active, are nearly present in industrial operations. These substances may have a decisive influence on the mass transfer and hydrodynamic processes. The interfacial tension is strongly affected by the surfactants and the internal circulation in the droplet is suppressed by surface-active agents,

which produce surface gradients of interfacial tension for a moving drop. The presence of surfactants has an effect on the system hydrodynamics. Accordingly, surfactants have a considerable effect on the terminal velocity and a number of workers have noted a strong influence of the system purity on terminal velocity or drag coefficient [2]. Industrial systems are rarely pure and surfactant accumulates at drop surface. The effect of surface-active agents can be summarised as: reduction of mass transfer coefficients, hindrance of drop circulation and oscillation, reduction of terminal velocity and reduction of any interfacial turbulence. The first theoretical approach to the effect of surface active materials on the velocity profiles around liquid drops was done by Levich [3]. It was shown that the surface-active materials increase the drag coefficient of the gas bubbles [4] and consequently reduce the mass transfer rate [5,6]. The possible interaction of surface active agents with mass transfer process may in general include both hydrodynamic and barrier effect, this subject has been extensively studied and is reviewed elsewhere [7].

The present work aims to predict the mass transfer rate from single liquid drop in the presence of two different types of surfactants non-corrosive, non toxic, namely Sodium Dodecyle Sulphate (SDS) and Carboxy Methyl Cellulose (CMC) in a spray column.

To this end, the system acetic acid/ toluene/water was chosen to conduct the present study with solute transfer direction from the continuous phase to the dispersed phase and vice versa. Comparison of the measured values of the MTC in the presence and in the absence of surfactants has been made to show the validity of single liquid drop data for use in column design work.

2. Experimental

2.1. Single drop apparatus

The experimental apparatus is almost the same as was used in the previous work [8,9] and will be discussed here only briefly.

A schematic diagram of the apparatus is shown in fig. 1, main parts of the apparatus consisted of perspex glass extraction column 0.15 m diameters, 0.7 m height. The column diameter was large enough to prevent wall effects as recommended by Grace [10].

The device applied for the formation of drops of a desired size was a glass capillary with a suitable diameter. The rising time of the drops through given distance was measured with a stopwatch. Solvent was 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 from the syringe setting and counting the number of drops formed in unit time [11]. Drops were generally spherical, moving without oscillation. Drops were formed at a reasonable rate, to ease counting and avoid interaction of drops with each other. The drops formed a layer on the water surface and this was removed by suction at regular intervals. Except otherwise mentioned all the experiments were conducted with the aqueous phase continuous and the organic phase dispersed at laboratory ambient temperature 25 ± 2°C. Reservoirs, pumps, valves and flow meters were provided to control and measure the flow of the continuous (aqueous) and dispersed (organic) phase.
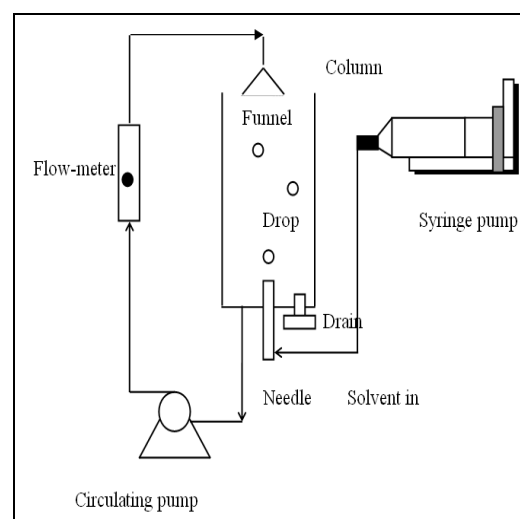

Fig. 1. Experimental set up.

2.2. Procedure

The column was filled with the aqueous phase (water) before the solvent (dispersed phase) commenced. Rotameters on the lines to and from the column enabled accurate flow settings and adjustments to be made thus ensuring that balanced flows were rapidly achieved and then maintained. A positive displacement syringe pump was used to introduce the organic phase into the bottom of the column. The pump and syringe were mounted on a higher level in order to avoid back flow of solvent in the syringe. After each experiment the column were thoroughly cleaned with Decon 90 solution and rinsed thoroughly [12]. Errors were minimised by measuring the rise time of 20 drops and the formation time of at least 50 drops were timed for each run, after a period allowed to reach a steady state conditions. The droplet rise velocities were determined by timing the rising time through a distance of 0.3 - 0.5 m, the bottom mark being approximately 0.05 m above the needle tip. The relative standard deviation on mean rise time is about 10 % and the average drop size measurement suffer an errors of about 2% incurred principally by uncertainty in counting the drops. The free rise experiments were repeated occasionally to check for the variation in contamination level.

All the chemicals used were of analytical grade reagent and the physical properties of the mutually saturated phases are given in table 1. In all experiments the inlet and outlet solute concentrations were determined by using spectrophotometer (UV-1601 Shimadzu) and some times by titration.

2.3. Materials and systems

The system studied was toluene - acetic acid-water [13,14]. The transferring solute was acetic acid, the continuous phase was water and the dispersed phase solvent was toluene. The physical properties were measured at 25°C ± 2 in the absence of surfactants. Mass transfer studies were carried out for acetic acid (solute) transfer from toluene (dispersed) to water (continuous phase). The distribution coefficient of

acetic acid is very high in favour of water, so the mass transfer resistance in this case was almost entirely due to the toluene (dispersed) phase.

Two surface active agents were placed, in turn, in the aqueous phase of each system namely; Sodium CMC and SDS. Sodium CMC [15] is a white, granular, oderless, tasteless powder, though it is generally produced and sold as solution of varying concentration and viscosity, it is formula is $[C_6H_7O_2(OH)_2COONa]_x$.

A solution of CMC and SDS with different initial concentrations ranged from 100 to 500 ppm were used. The preparation of solutions were done by spreading the desired amount of surfactants powder over the top water surface. The temperature of all solutions was the ambient temperature 25°C ± 2. Except otherwise mentioned the concentration of SDS or CMC in the aqueous solution was 400 ppm for all other experiments. It has to be noted that the concentration range considered here is below the critical micelle concentration.

3. Results and discussion

The overall mass transfer coefficients for drops of known size may be measured 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. Collection of solvent drops in a glass funnel, covering about a quarter of the cross-section area of the column, was found satisfactory [16]. The funnel was immersed to an extent such that the interface where drops coalesced was in the funnel stem [13]. 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:

Table 1
Physical properties of the system used

ρ_c (kg/m³)	ρ_d (kg/m³)	μ_c (kg/ms.10³)	μ_d (kg/ms.10³)	γ (mN/m)
Systems : Toluene/acetic acid (5%)/water				
995	866	0.87	0.55	21.35
Systems : Toluene/acetic acid (5%)/water				
Solute transfer from continuous to dispersed phase				
100	858	0.89	0.54	21.5

$$-V \, dC / dt = K_{od} A_d (C - C^*). \qquad (1)$$

Integrating eq. (1) yields:

$$(C_o\text{-}C_i) \ / \ (C^* \text{-} C_i) = (1 \text{-} exp \ (\text{-} \ 6 \ K_{od} \ t \ /d)). \quad (2)$$

$$K_{od} = \text{-} \ (d \ / \ 6 \ t) \ ln \ (1 \text{-} E). \quad (3)$$

Where $E = (C_i \text{-}C_o)/(C_i \text{-} C^*)$ and C_i is the inlet and C_o outlet solvent concentration at the top collection point, and since there was no solute initially in the water, the drop concentration in equilibrium with the aqueous phase, $C^* = 0.0$. Hence the overall droplet mass transfer coefficient may be evaluated by knowing the drop size, the drop rise time and the measured solute concentrations.

Figs. 2 and 3 show the influence of surfactant concentration on the mass transfer coefficient. The surfactant is found to be effective on smaller droplets, the droplet of more total amount of the same surface active agents have slower internal motion. Ishii [4] reported that the primary effect of surfactant appeared to be on droplet hydrodynamic and particularly on oscillation, turbulence and internal circulation in droplets. It is clear from figs. 2 and 3, as the concentration of surfactant was increased, a marked decrease in MTC was observed for drops and this usually

Fig. 2. Effect of surfactant (SDS) concentration on the mas transfer coefficient.

accompanied by earlier boundary layer separation and irregular drop oscillations

[6]. At relatively high surfactants concentration (> 500 ppm) the systems were said to be grossly contaminated. In all cases the addition of SDS and CMC resulted in a higher drag coefficient [4, 17]. The magnitude of this difference increases with increasing solute concentrations. Moreover, Sedahmed et al. [18] have shown that the polymer addition decreases the mass transfer coefficient by an amount ranging from 7.5 to 51 % depending on polymer concentration and the degree of stretching of polymer molecules with a consequent increase in their effectiveness in damping small-scale, high intensity eddies. The action of these substances in a drop liquid dispersion is complicated by the fact, when the frequency of drop formation is high and new substances are formed at high rate, surface equilibrium may not necessarily be established. Consequently the concentration of the adsorbate at the interface would vary with time this effect would be time dependent.

For comparison between SDS and CMC a better picture of the effects of surfactant type can be obtained by relating the drop formation time to equivalent drop diameter at constant concentration of surfactants as shown in figs. 4, 5 and 6.

It is clear that SDS increase formation time of the drop compared to the values obtained by using CMC. The reason for this discrepancy may be due to that SDS is a high molecular weight compound (M.Wt. 288) and is very soluble in water but less or insoluble in organic solvents [19]. When SDS is dissolved in water the
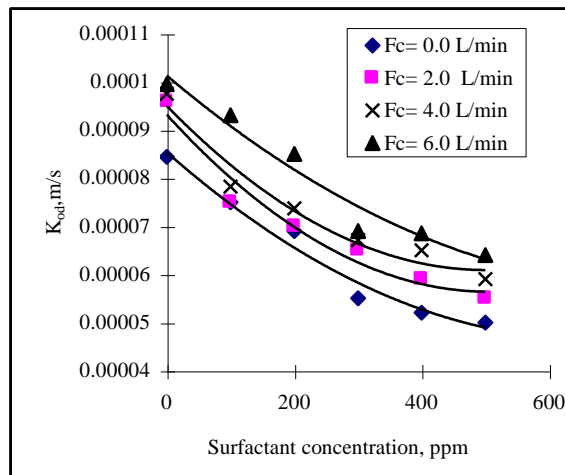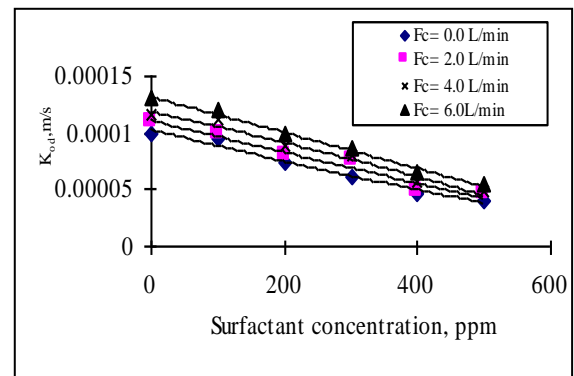

Fig. 3. Effect of surfactant (CMC) concentration on the mass transfer coefficient.
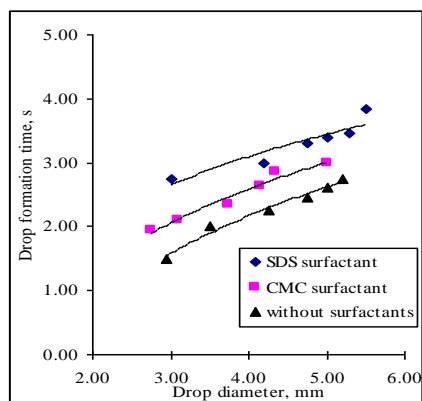
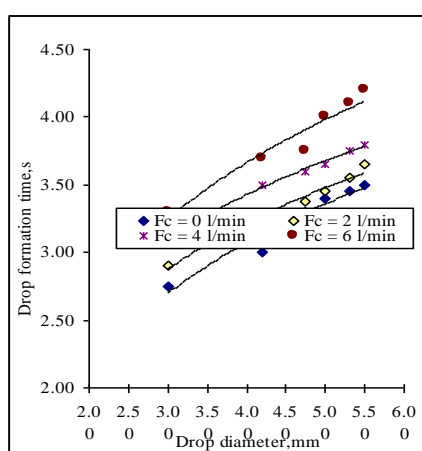Fig. 4. Drop formation time versus drop diameter in the presence and in the absence of surfactants.

Fig. 5. Drop formation time versus drop diameter in the presence SDS surfactant at different flow rate.
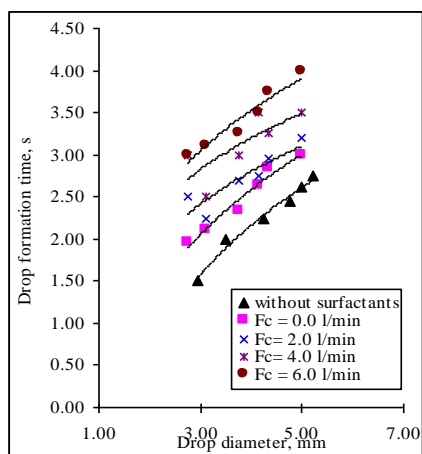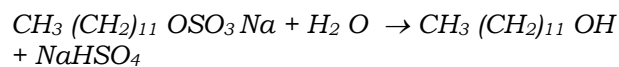

Fig. 6. Drop formation time versus dropdiameter in the presence of CMC surfactant at different down flow rate.

following reaction, with the formation of highly surface-active dodecanol (dodecyl alcohol), takes place [20]:

$$CH_3 (CH_2)_{11} OSO_3 Na + H_2 O \rightarrow CH_3 (CH_2)_{11} OH + NaHSO_4$$

Dodecanol is a non-polar organic molecule, which is highly soluble in organic solvents but quasi insoluble in water [21]. The energy of adsorption of surface-active impurities is relatively low. Thus, while solvent such as benzene and toluene are very easily contaminated, drops of butyl acetate in water are much less readily affected by surface-active impurities. CMC is a low molecular weight (M Wt. 212) surfactants, need a higher concentration to decrease the fraction loses while SDS surfactants show a great reduction at low concentration compared to that of CMC. The reason is mainly due to the molecular weight effect and the length of the carbon chain, in which there is a strong correlation between the effectiveness of drag reduction and the molecular weight of surfactants. Additionally, the higher the degree of polymer stretching the higher its ability to damp the small scale high frequency eddies which prevail in the boundary layer.

However, as has already been pointed out, the addition of any surface active substance to water-drop system would be expected to cause considerable alterations in the flow behaviour. Surface active substances have been observed to lower the terminal velocity of drops, increase the drag at given drop size, cause drop to be more spherical than those in water at the same size, and decrease interfacial as well as internal turbulence [21]. The combination of these effect should result in change from spheroidal to spherical drop occurring at a larger size in the presence of surface active solute than in distillate water. The size at which this transition occurs should depend upon the concentration and the surface activity of the substance. In parallel, Sadd et al. [22] showed that surfactants are capable of displaying very high mass transfer resistance which increase rapidly with the length of the surfactants carbon chain.

Fig. 7 shows the extraction percentage, defined as (inlet solute concentration – outlet solute concentration/inlet solute concentration or C_i-C_o/C_i) against drop size. It is clear that the

percentage of extraction in the presence of surfactants is less than that without surfactants.

Figs. 8 and 9 show a comparison between the reduction in the MTC in the presence and absence of surfactants. It is evident that the presence of surfactant decreases the mass transfer coefficients and SDS causes a higher reduction in the mass transfer coefficients even at low concentration compared to that of CMC. This is mainly due to the molecular weight effect and the length of the carbon chain of SDS [15, 19].

Many investigators have observed a reduction in mass transfer rate in the presence of surface-active substances [2, 23]. Several explanations have been proposed to describe the phenomenon. The reduction in mass transfer has been explained resulting from the partially or completely suppressed internal circulation inside drops [24]). Levich [3] proposed the idea that the presence of surface active species gives rise to a concentration gradient on a mobile drop surface. This concentration gradients gives rise to an interfacial tension gradient or a force acting in the surface to oppose drag force on a moving drop. Thus internal circulation of drop contents may be reduced; hence local mass transfer coefficients are smaller. An alternative analysis has been presented by Karsmanev [25,26]. Who assumed that a stagnant cap forms over the rear of the droplet as surface active substance are added to the system. This cap tend to enlarge with increasing initial concentration of surface active agent. This simple model divides the drop surface into two distinct and uniform regions, one covered with surface-active agent and the remaining free of it. Since the surface velocity in the first region is zero, the mass transfer will be retarded. Calculation shows that adding to water a small quantity of SDS or CMC surfactants (200 ppm) reduce the mass transfer coefficients by 20 to 50 % depending on the velocity of the continuous phase and type and concentration of surfactants used as shown in figs. 7, 9, 10.
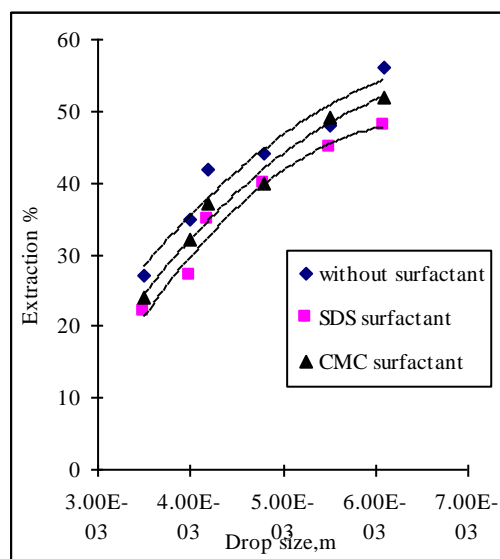

Fig. 7. Extraction % as a function of drop size in the presence and in the absence of surfactants.
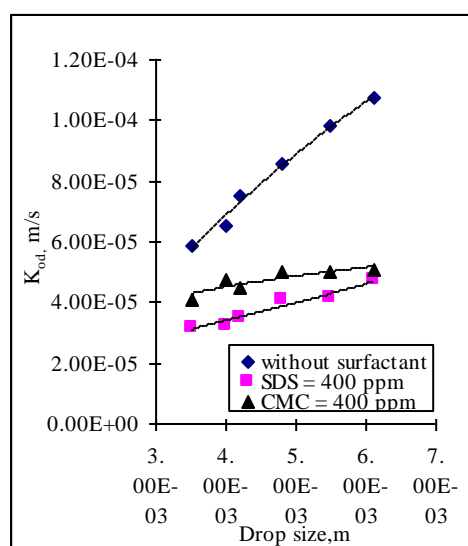

Fig. 8. The mass transfer coefficient as a function of dropsize in the presence and in the absence of surfactants.

The effect of the continuous phase velocity on the mass transfer coefficient in the presence of surfactants was found to be very significant with only a slight increase in the drop size occurring with increasing the down flow of the continuous phase.

The effect of down flow on the dispersed phase mass transfer coefficient is associated directly with movement of drops through the column.

Other important effects are obtained by reducing drop relative velocities [26] and increasing in contact time leading to reduction in k_d and a change in the product $k_d t / d$ which affect the fraction extraction.
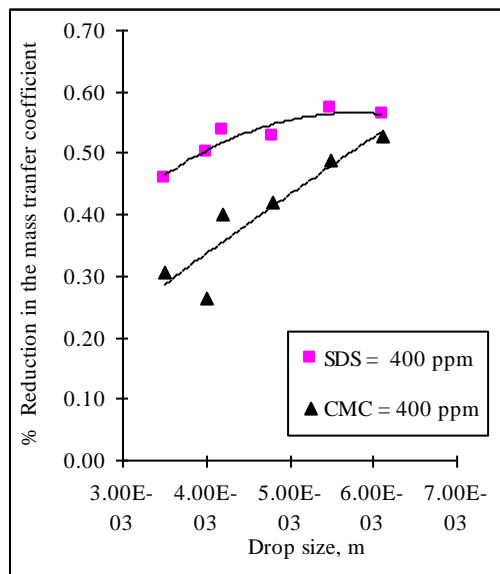

Fig. 9. The percentage reduction in the mass transfer coefficient as a function of drop size in the presence of surafactants.
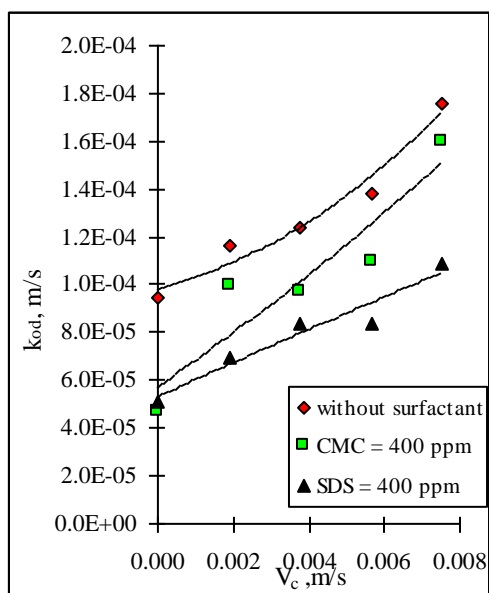

Fig. 10. Effect of continuous phase velocity on the mass transfer coefficient in the presence and on the absence of surfactants.

In liquid-liquid extraction operations, the dispersed phase possess a rather wide range of drop size. The large drops pass through the column more rapidly than do drops of average size, whereas the small ones reside for a longer time and may be highly backmixed owing to their inertia. Consequently, there exists a wide distribution of drop residence time. Drop size are known in this experimental work, they depend on needle diameter, slightly on rotameter flow rate [27,28].

Fig. 10 displays the profile of down flow of the continuous phase expressed as the continuous phase velocity against the mass transfer coefficient in the presence and in the absence of surfactants. The mass transfer coefficient was found to increase with increasing the velocity of the continuous phase for of surfactants free – solutions, [29]. Fig. 10 also shows that the mass transfer coefficient decreases in the presence of surfactants. As mentioned before, surface-active substances tend to accumulate at the interface between two fluids, thereby reducing the surface tension. When a drop moves through a continuous medium, adsorbed surface active materials are swept to the rear leaving the frontal region relatively uncontaminated [30]. The concentration gradients results in a tangential gradient of surface tension which in turn causes a tangential stress tending to retard surface motion. These gradients are most pronounced for small fluid particles to be particularly subject to retardation [31,32].

Fig. 11 show the dependence of the average mass transfer coefficient on drop size and direction of solute transfer. The coefficients for mass transfer direction from the dispersed phase into the continuous phase ($d \rightarrow c$) are slightly higher than that for the mass transfer from the continuous into the dispersed phase ($c \rightarrow d$) in the presence and in the absence of surfactants. 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.

It is well known that surface instability or Marngoni effect can increase solute transfer rates appreciably. 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
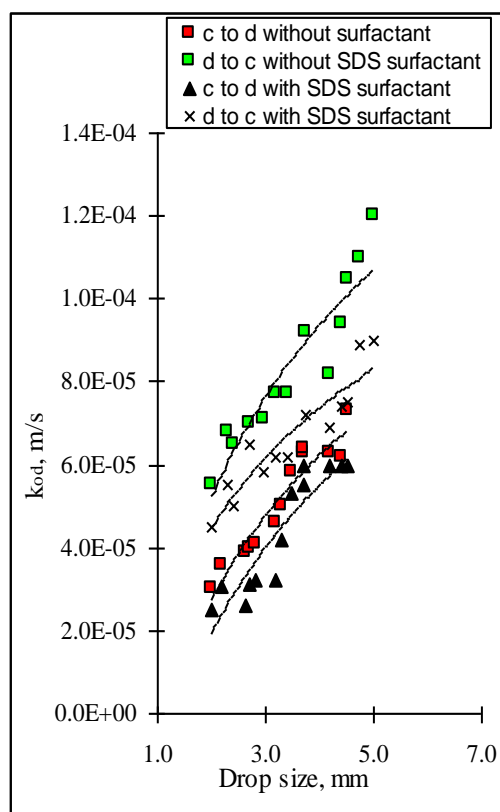
Fig. 11. The mass transfer coefficient as a function of drop size in the presence and in the absence of surfactants at different direction of solute transfer

transfer. With the present liquid systems, drop instability and tendency for drop coalescence is enhanced when the solute is transferred from the dispersed phase to the continuous phase and the drops are stabilised for the opposing direction of solute transfer. These effects have been onfirmed in experiments on inter- drop coalescence in spray column [33].

5. Conclusions

Mass transfer measurements have been made for single liquid drops rising in a stationary and moving phase in the presence of SDS and CMC surfactants in a spray column. From these results, combined with the previous work, some conclusions can be drawn about the influence of these surfactants on mass transfer coefficient as follows:

The effect of trace amount of surfactants on all stages of drop is pronounced. The surfactant solution lengthen the lifetime of the droplet. Minute quantities of some surface active substances exert profound and manifold effect on the behaviour of drop in water even in extremely dilute solutions, These substances have a pronounced effect on both the hydrodynamic and on the rate mass transfer from rising the drops. The mass transfer coefficient decreases with increasing the concentration of surfactants.

It was found that surfactant decreases MTC by an amount ranging from 20 to 50 %depending on type of surfactant added. Also, the relative velocity between drops and the down-flowing continuos phase, in the presence or absence of surfactants, were found to decrease as the down flow rate increased. The mass transfer coefficient was found to increase with increasing the velocity of the continuous phase for solution free of surfactants.

The mass transfer coefficients in purified systems (surfactants free solution) are higher than that with surfactants. It is concluded that CMC has a significance effect on the value of MTC, but the effect of SDS is higher.

The decrease in the mass transfer coefficient upon the addition of surfactants is not the results of a surface resistance, but is primary due to the changes in the hydrodynamic characteristic of the system. Surface active contaminants play an important role in damping out internal circulation in deformed drops. However, there are indication that deformations tends to decrease internal circulation velocities significantly while shape oscillations tend to disrupt the internal circulation pattern of droplets and promote rapid mixing.

The coefficients for mass transfer direction from the dispersed phase into the continuous phase $(d \rightarrow c)$ are slightly higher than that for the opposite direction $(c \rightarrow d)$. in the presence and in the absence of surfactants. The transfer from the dispersed to the continuous phase promotes droplets coalescence thus increasing mean drop size.

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Nomenclature

C_i, C_o is the inlet and outlet solute conc, ppm,

$c \to d$ is the continuous to dispersed phase solute direction,

d is the drop diameter, m,

$d \to c$ is the dispersed to continuous phase solute direction,

E is the degree of extraction during rise period, $(C_i - C_o)/(C_i)$,

F_c is the down flow of the continuous phase, l/min,

k_c, k_d is the contin. and disp. phase mass transfer coefficient m/s,

K_{od} is the overall mass transfer coefficient, m/s,

t_f, t_r is the drop formation time, drop rise time respectively, s, and

V_c, V_d is the continuous phase velocity, dispersed phase velocity, m/s.

Greek letters

μ Viscosity, kg/m s,

ρ Density, kg/m^3,

$\Delta\rho$ Phase density difference kg/m^3, and

γ Interfacial tension mN/m.

Subscripts

c Continuous, and

d Dispersed.

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