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Soil Cleanup by In-Situ Surfactant Flushing. VII. Determination of Mass Transfer Coefficients for Reclamation of Surfactant for Recycle

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ABSTRACT

Mass transfer coefficients were determined for the extraction of naphthalene in 50 and 100 mM aqueous sodium dodecylsulfate (SDS) solutions (the continuous phase) into hexane (dispersed phase). The effect of surfactant was explored in a series of single drop experiments. Mass transfer coefficients determined experimentally fall between the values predicted by correlations for circulating and non-circulating drops. The presence of SDS does appear to reduce the mass transfer coefficients as compared to those for pure water.

INTRODUCTION

The contamination of soils and groundwater with volatile and/or nonvolatile organics from underground storage tanks, spills, and improper waste disposal presents a major remediation problem in the United States and other industrial nations. The removal/destruction of organics either in the absorbed state or present as dense nonaqueous phase organics (DNAPL) has been approached using several technologies: pump and treat, soil vapor stripping, in-situ biodegradation, in-situ heating using radio frequencies, surfactant flushing, and others. This paper is concerned with surfac-

tant flushing. Ellis et al. (1) were among the first to publish results of a lab-scale study on surfactant flushing. Nash (2) performed a field study of surfactant flushing on a small scale. Vigon and Rubin (3) examined surfactant selection and optimal dosage requirements. Our group has published several experimental and theoretical aspects of surfactant flushing (4–11).

Surfactant flushing removes organic contaminants from soil and groundwater by solubilizing them within micelles in the surfactant solution. This solubilization makes surfactant flushing much more efficient than flushing with water alone when attempting to remove hydrophobic organic contaminants, e.g., DNAPLs. Underwood et al. (11) focused on the problems associated with spent surfactant treatment and surfactant recycle. For surfactant flushing to be economical, it is necessary to recycle the surfactant and efficiently remove the contaminants. An anionic surfactant (sodium dodecylsulfate, SDS) was chosen so that solvent extraction could be used to remove the contaminants and reclaim the surfactant solution for reuse. We believe that anionic surfactants would be much less soluble in nonpolar solvents than nonionic surfactants, making solvent extraction possible. Gannon et al. (6) showed that gentle extraction of *p*-dichlorobenzene (DCB), and naphthalene from SDS solution into hexane was possible. Underwood et al.'s (11) results showed that extraction of contaminated SDS solutions with hexane was an effective method for cleaning up these solutions for recycle. The next step in the development of a reclamation process for the recycle of surfactant solutions is the development of design and scale-up information. In this work we determine the continuous-phase mass transfer coefficients for the extraction process used to reclaim the surfactant solution and remove the contaminants. Mass transfer coefficients are determined for two different surfactant concentrations and also for systems having no surfactant present. These studies help to determine the effect of surfactants on the mass transfer process.

Mass Transfer into Drops

The transfer of a solute from a continuous phase to a dispersed phase has been studied by several researchers (12–18). The opposite case of mass transfer from the dispersed phase to the continuous phase has also been investigated (19–25). All of these researchers attempted to model the mass transfer occurring between a single drop and a continuous phase. The general idea was that the models of the single drops could be used to approximate the mass transfer occurring in a spray or perforated-plate column in which a continuous phase is in contact with many drops. Corre-

lations for mass transfer coefficients have been developed, and the fractional extraction achieved in columns has been determined.

Researchers have concentrated on studying three periods of time in the single drop experiments in which mass transfer takes place: 1) formation of the drops in the continuous phase, 2) free rise or fall of the drops through the continuous phase, and 3) coalescence of the drops at the end of the free-rise/fall period (15). This study focuses on the mass transfer taking place during the free-rise period. Correlations are presented that relate the Sherwood number (containing the mass transfer coefficient) to the Reynolds and Schmidt numbers. Correlations are given for both circulating and rigid drops. The effect of surfactants on mass transfer is discussed and the results compared to previous studies.

Free-Rise Period

The system studied in this project consists of hexane drops as the dispersed phase and an aqueous solution of SDS as the continuous phase. Mass transfer of organic solutes (phenanthrene, naphthalene, and biphenyl) is from the continuous to the dispersed phase. Previous work (13) indicated that the distribution of *p*-dichlorobenzene (similar to the compounds used in this study) between the hexane and SDS phases heavily favored the hexane phase. The resistance in the hexane phase is thus considered to be negligible compared to the continuous (SDS) phase resistance. For this reason the mass transfer coefficient correlations given later are for continuous-phase resistance.

The continuous-phase mass transfer coefficient, k_c , can be calculated for the free-rise period of a drop. An assumption is made that the mass transfer occurring during drop formation is small due to rapid drop formation. The mass transfer during coalescence of drops is minimized by continually drawing off coalesced drops. Mekasut et al. (17) devised a way of calculating k_c from the overall dispersed-phase mass transfer coefficient, K_D . The differential material balance for a drop during its steady rise is

$$\frac{d(VC)}{dt} = K_D(C^* - C)A \quad (1)$$

The drop volume V and equilibrium concentration of the solute in the drop C^* are assumed to be constant. Equation (1) is integrated to give

$$K_D = \frac{-V}{A(t_{\text{rise}})} \ln \frac{C^* - C_{\text{out}}}{C^* - C_{\text{in}}} \quad (2)$$

where V = volume of a sphere with the same diameter as the hexane drop (cm^3)

A = surface area of a sphere with the same diameter as the hexane drop (cm^2)

t_{rise} = rise time of a hexane drop in the column (s)

C^* = equilibrium concentration of contaminant in hexane (mg/L)

C_{out} = concentration of contaminant in hexane at the outlet of the column (mg/L)

$C_{\text{in}} = 0$ = concentration of contaminant in hexane at the inlet of the column (mg/L)

The relationship between K_D , k_d , and k_c is

$$\frac{1}{K_D} = \frac{1}{k_d} + \frac{m}{k_c} \quad (3)$$

in which m is the distribution coefficient and k_d is the local dispersed-phase mass transfer coefficient. If k_d is assumed to be of the same order of magnitude as k_c and m is large, then $1/k_d \ll m/k_c$ and Eq. (3) becomes

$$k_c \approx mK_D \quad (4)$$

Treybal (26) gives correlations for the Sherwood number for rigid spheres and circulating drops. The limiting case for the smallest rates of transfer would be the rigid sphere case. Treybal (26) gives a correlation for rigid spheres developed by Steinberger and Treybal (27):

$$\text{Sh} = \frac{k_c d_p}{D_c} = \text{Sh}' + 0.347(\text{ReSc}^{0.5})^{0.62} \quad (5)$$

where Sh' , which accounts for both natural convection and molecular diffusion in completely stagnant fluids, is given by

$$\text{GrSc} < 10^8: \text{Sh}' = 2 + 0.569(\text{GrSc})^{0.250} \quad (6)$$

$$\text{GrSc} > 10^8: \text{Sh}' = 2 + 0.0254(\text{GrSc})^{1/3} \text{Sc}^{0.244} \quad (7)$$

In the above correlations, the following terms can be defined:

k_c = mass transfer coefficient in the continuous phase (cm/s)

d_p = diameter of a sphere of volume equal to that of a drop (cm)

D_c = molecular diffusivity of the solute in the continuous phase (cm^2/s)

Re = Reynolds number = $d_p V_t \rho_c / \mu_c$

ρ_c = density of the continuous phase (g/cm^3)

μ_c = viscosity of the continuous phase (P)

Sc = Schmidt number = $\mu_c / \rho_c D_c$

Gr = Grashof number = $gd_p^3\Delta\rho\rho_c/\mu_c^2$

v_t = terminal velocity of a drop (cm/s)

$\Delta\rho$ = difference in densities in the continuous phase between liquid at the sphere surface and in the main bulk of the continuous phase (g/cm³)

Garner et al. (19) also give a correlation for mass transfer involving organic spheres

$$Sh = 2 + 0.95Re^{1/2}Sc^{1/3} \quad (8)$$

Garner and Skelland (28), however, observed that fluid droplets differ from rigid spheres in three ways during free fall: 1) the frictional drag of the surrounding fluid may induce circulation of both the interface and interior of the droplets; 2) the drop may depart from spherical shape, assuming some oblateness, due to gravity; and 3) a prolate-oblate type of oscillation may occur.

Treybal (26) gives two correlations for circulating drops. For the continuous phase in potential flow ($\mu_c = 0$) and large Peclet numbers:

$$Sh = 1.13Pe^{1/2} = 1.13 \left(\frac{d_p v_t}{D_c} \right)^{1/2} \quad (9)$$

where Pe is the Peclet number which equals the Reynolds number times the Schmidt number. The second correlation for circulating drops given by Treybal (26) is for Pe in the range 3,600 to 22,500:

$$Sh = 5.52 \left(\frac{\mu_c + \mu_d}{2\mu_c + 3\mu_d} \right)^{3.47} \left(\frac{d_p \sigma \rho_c}{\mu_c^2} \right)^{0.056} Pe^{1/2} \quad (10)$$

Garner et al. (19) correlated their data for mass transfer in circulating drops with

$$Sh = -126 + 1.8Re^{1/2}Sc^{0.42} \quad (11)$$

Thorsen and Terjesen (14) used a similar correlation to fit their data:

$$Sh = -178 + 3.62Re^{1/2}Sc^{1/3} \quad (12)$$

They found Eq. (12) to be equally applicable to noncirculating drops as to circulating drops. Thorsen and Terjesen (14) also remark that their results obtained with systems having interfacial tensions about 40 dyn/cm also fit the correlation of Garner et al. (19), Eq. (11), which was derived for systems with low interfacial tensions between 3 and 7 dyn/cm. Garner et al. (19) and Thorsen and Terjesen (14) present differing explanations of the mechanism of mass transfer involving liquid drops.

Garner et al. (19) proposed that the mass transfer rate through the continuous phase film of circulating drops is enhanced by the thinning of the boundary layer induced by the circulation. This boundary layer thickness is assumed to be between that for a solid sphere and that for a sphere in potential flow ($\mu_c = 0$).

Thorsen and Terjesen (14) believe that internal circulation and mass transfer are two different and largely unconnected phenomena associated with the fluid boundary. They do accept the idea that internal circulation leads to a thinning of the boundary layer, but they believe that this effect is insufficient to account for the great differences between the rates of mass transfer for drops and solid spheres. Thorsen and Terjesen (14) state that interfacial agitation occurs when a solute is transferred across the liquid-liquid interface. Their studies indicated that high rates of mass transfer cannot be explained by interfacial agitation. They proposed the idea that the ring vortex formed at Reynolds numbers greater than about 20 produces disturbances in the unstable fluid boundary region, and that the high rates of mass transfer appear as a result of these hydrodynamic disturbances. They call this hydrodynamic effect interfacial turbulence. They cite that it is well known in hydrodynamics that an unstable boundary region is formed between two liquid streams having velocities which differ in magnitude or direction. Thorsen and Terjesen (14) also think that mass transfer occurs mainly at the rear of the drop due to the turbulent motion in the continuous phase close to the boundary.

Effect of Surfactants on Mass Transfer

Several researchers have observed the effects of the presence of surfactants on mass transfer to liquid drops. The way in which surfactants affect mass transfer may be divided into hydrodynamic and physicochemical effects (17, 29). The important hydrodynamic effects are changes in circulation velocities, reduction of rise velocity, and the restriction of interface movement because of interfacial tension gradients. The physicochemical effects are surface blocking or interactions between solute and surfactants.

Beitel and Heideger (21) note that there are two conflicting views as to the distribution of surfactant over the surface of a moving drop. The first view considers that surfactant molecules, as they are adsorbed, are continuously swept toward the rear of the drop by the movement of the fluid interface. Surfactant molecules accumulate at the rear of the drop with a nonuniform distribution over the interface with concentration highest at the rear and gradually decreasing toward the front. A gradient in interfacial tension occurs due to the gradient in surfactant concentration. This inter-

facial tension gradient counteracts part of the shearing stress caused by drop movement through the continuous phase, thus reducing circulation in the drop.

The second view on the distribution of surfactant also assumes that surfactant molecules are swept to the rear of the drop by convective motion but accumulate in a dense monolayer growing forward from the rear stagnation point (21). This gives the drop a "spherical cap coverage." Beitel and Heideger (21) found that the cap tends to grow larger as the initial concentration of surfactant in the continuous phase increases. They proposed a model that divides a drop into two regions, one covered with surfactant molecules and another with no surfactant present. The surface velocity in the area covered with surfactant molecules is essentially zero; mass transfer then is reduced to that associated with a solid sphere.

Garner and Skelland (28) observed that surface-active impurities in their system retarded mass transfer and caused nitrobenzene drops to become stagnant. They postulated that there was an adsorbed film of surface-active material present on the drops. They studied this phenomena by adding SDS to a clean system. Garner and Skelland (28) assumed that the dodecyl chain lies flat near the plane of the interface and is a cylinder about 13.8 Å in length and 20.5 Å² in cross section.

Mekasut et al. (17) studied the effect of a surface-active agent, Teepol, on mass transfer of iodine from an aqueous phase to a falling drop of carbon tetrachloride. They found that the presence of Teepol increased drag coefficients and reduced the frequency of oscillation of the drops to some extent. They also observed that the fall velocity of the drops was reduced, and the mass transfer coefficient decreased by 58% in comparison with the pure system.

EXPERIMENTAL

Chemicals and Apparatus

The SDS (Fluka) used in the experiments was 98% pure. Other chemicals employed were hexane (Fisher, certified and spectranalyzed), phenanthrene (Eastman and Fluka), naphthalene (Fisher), biphenyl (Aldrich), sodium chloride (Fisher), and toluene (Fisher). All chemicals were used as received.

Extraction Studies with Single Drops of Hexane

A schematic of the set-up for the extraction experiments involving a stream of single hexane drops is given in Fig. 1. The experiments were performed in a glass column, 4.4 cm in diameter and 122 cm in length.

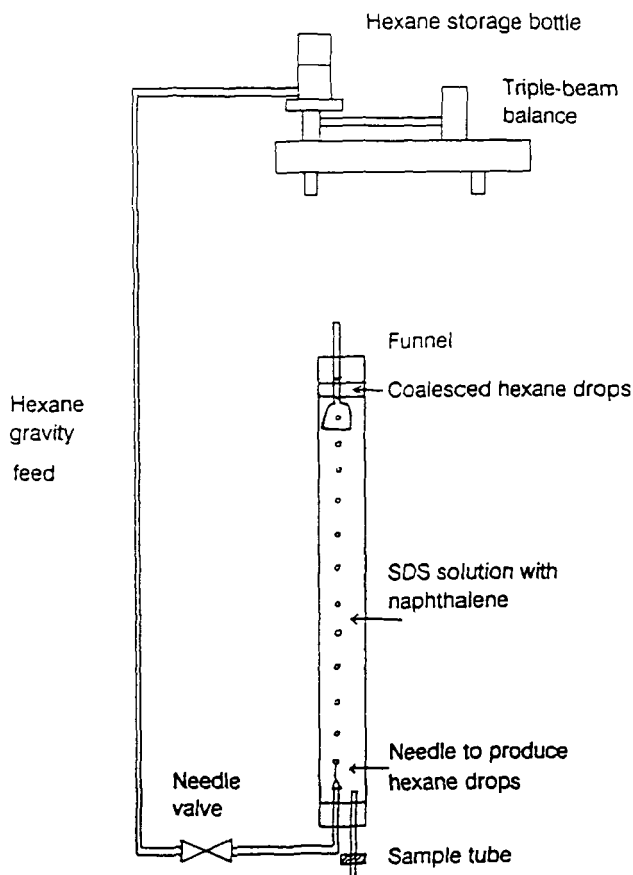


FIG. 1 Setup for the extraction experiments involving a stream of single drops of hexane.

The procedure was to fill the column with contaminated SDS solution or contaminated water to an average height of 110 cm (1750 mL). An inverted funnel was placed in a rubber stopper at the top of the column so that the top of the aqueous layer reached the neck of the funnel. A glass bottle with a spigot at the bottom was filled with hexane, placed on a triple-beam balance several feet above the column (for gravity feed), and its weight in grams was recorded. The needle valve was opened, and hexane drops were introduced into the column via a syringe needle or glass tube at the bottom of the column. The hexane flow rate was adjusted to the desired flow (65–80 drops/min), the weight of the bottle of hexane was recorded again, and timing of the run began. The height of the aqueous

phase in the column and the rise times of the drops were measured to determine the average rise velocity of the drops. The drop flow rate was checked every 10 minutes during a run by timing the drops with a stopwatch. Coalesced hexane drops collected in the neck of the funnel and were sampled (about 0.5 mL) every 5 or 10 minutes. A run was continued until 4 to 6 samples were collected (about 30–50 minutes).

An experiment was concluded by closing the needle valve, recording the weight of the bottle of hexane, and taking a sample of the aqueous phase (about 5 mL). The average drop diameter was calculated using the volume of hexane used during the run and the drop flow rate, and assuming the drops were spherical (hexane drops in water were rounder than those in the SDS solutions). The contaminant absorbance was determined by UV spectrophotometry (10). The hexane samples had to be diluted with fresh hexane to a volume of about 3 mL so they could be analyzed in the spectrophotometer.

The diameter of the hexane drops was varied from 0.10 to 0.20 cm in the SDS solutions and 0.2 to 0.3 cm in water. The aqueous media used were water, 10 mM SDS, and 50 mM SDS with a naphthalene concentration of about 20 mg/L, the solubility of naphthalene in water.

RESULTS AND DISCUSSION

The effect of surfactant on the mass transfer of naphthalene was explored in the extraction experiments with single drops of hexane. A stream of hexane drops was passed through a column of naphthalene-contaminated SDS solution, and continuous-phase mass transfer coefficients were calculated for each drop size, as pictured in Fig. 2. The k_c values for solutions containing no surfactant appear to be larger than those for 10 and 50 mM SDS solutions. This point is emphasized in Fig. 3 in which k_c is plotted versus SDS concentration for the same size hexane drops. Garner and Skelland (28) and Mekasut et al. (17) observed that surfactants decreased the mass transfer coefficients in their systems, impaired drop circulation, and reduced the rise velocity of drops. The velocity of hexane drops in this study as a function of hexane drop diameter is depicted in Fig. 4. The rise velocities in water for hexane drops about 0.2 cm in diameter are reduced by about 30% in the SDS solutions. The hexane drops in water were also more spherical in shape than were the rather elliptical drops in the SDS solutions. The drops in the SDS solutions exhibited a maximum in drop rise velocity at diameters of about 0.155 cm. The mass transfer coefficient in Fig. 2 also increased up to drop diameters of about 0.16 cm, although the data were somewhat scattered. This phenomenon was also observed by Garner et al. (19) in the presence of no surfac-

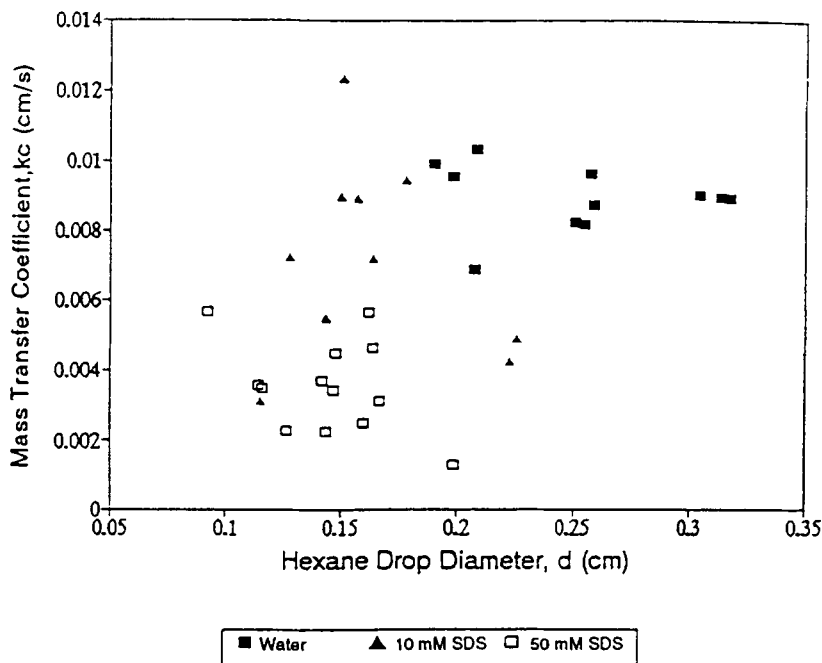


FIG. 2 The continuous-phase mass transfer coefficient of naphthalene as a function of hexane drop diameter.

tant. They suggested a hydrodynamic correspondence for the maxima in their k_c versus d curves. In this study, drops larger than about 0.16 cm were somewhat elliptical in shape and periodically oscillated, resulting in a decrease in drop velocity. The percent decrease in k_c (52% in 10 mM SDS, 68% in 50 mM SDS) with increasing drop diameter corresponds to the drop rise velocity squared.

Garner et al. (19) and Thorsen and Terjesen (14) described the mass transfer of solutes into drops. They believe that one factor contributing to mass transfer is internal circulation in drops that reduces the boundary layer thickness through which solutes have to diffuse. Beitel and Heideger (21) believe that mass transfer in the presence of surfactants is reduced due to a collection of surfactant molecules on the solvent drops that reduces drop circulation. In the present extraction studies, drop circulation was difficult to observe, but the observed mass transfer coefficients could be compared to correlations for k_c for both circulating and noncirculating drops. The diffusion coefficient for naphthalene in water was estimated using the Wilke–Chang correlation (30) to be 6.66×10^{-6} cm²/s. Due to

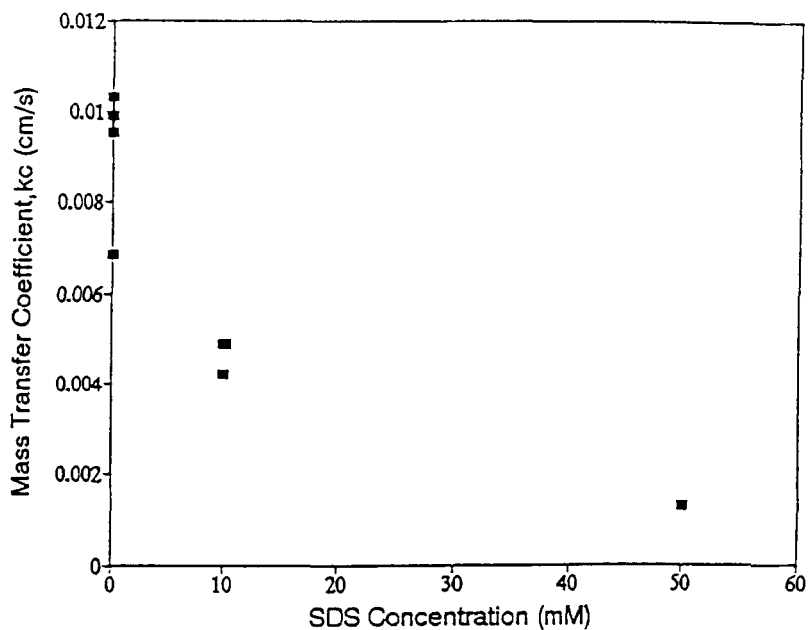


FIG. 3 Comparison of mass transfer coefficients in water, 10 mM SDS, and 50 mM SDS for a hexane drop diameter of 0.2 cm.

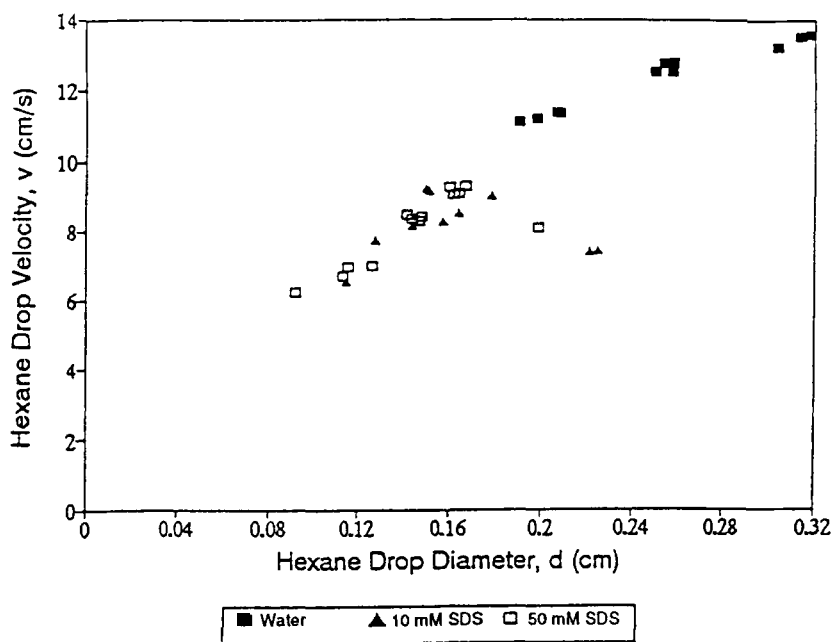
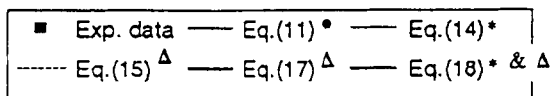
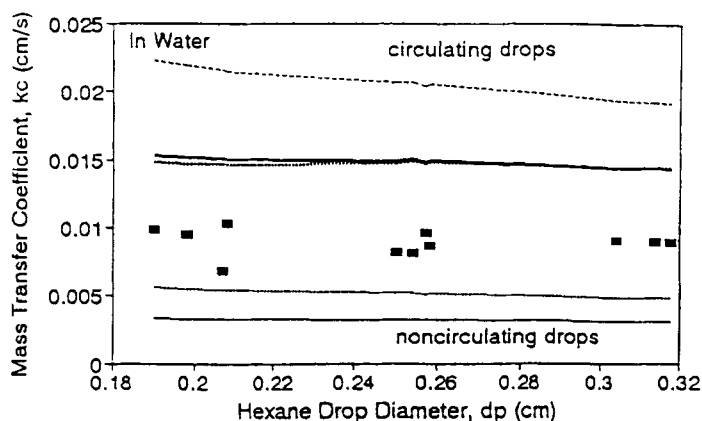


FIG. 4 Hexane drop velocity as a function of hexane drop diameter.

limited data on diffusion coefficients in surfactant solutions, the diffusion coefficients for naphthalene in SDS solutions were assumed to be the same as its diffusion coefficient in water. Figures 5–7 compare the experimentally determined k_c values with correlations for k_c , Eqs. (5), (8), (9), (11), and (12). Figure 5 shows the mass transfer coefficient for naphthalene in water, two correlations for noncirculating drops, two correlations for circulating drops, and one correlation that is believed to apply to both circulating and stagnant drops (14). The experimental data in water fall between the correlations for circulating and noncirculating drops. Figure 6 pictures the k_c data in 10 mM SDS and k_c values calculated using the previously mentioned correlations. Most of the experimentally determined k_c values fall between the k_c values for circulating and noncirculating drops with some of the smaller and larger drops sizes falling



* noncirculating

Δ circulating

* & Δ circulating and noncirculating

FIG. 5 Comparison of experimental k_c 's to correlations for k_c in water.

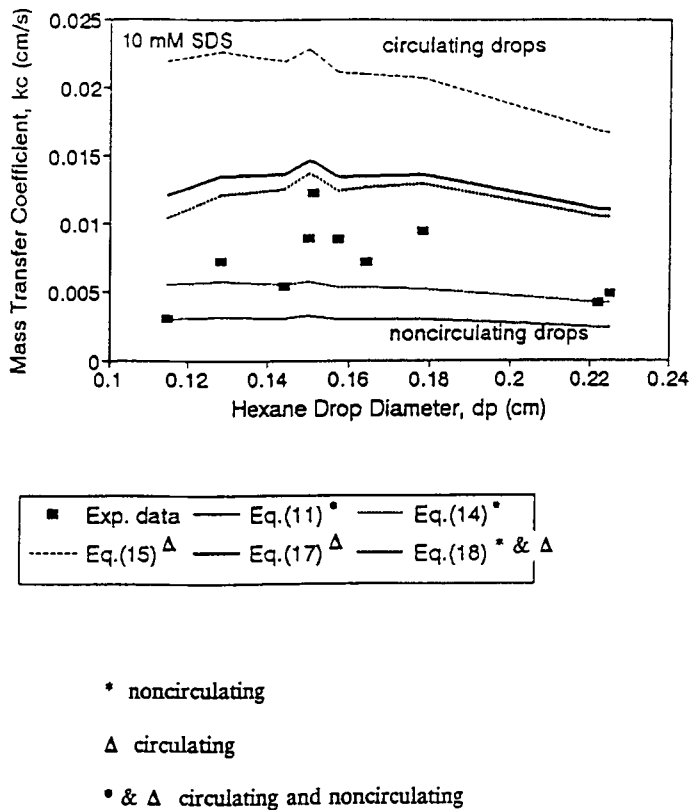
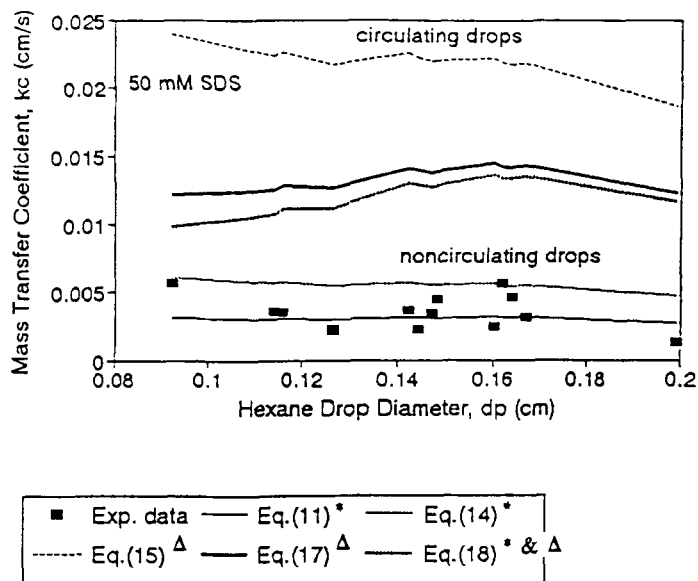


FIG. 6 Comparison of experimentally determined k_c values to correlations for k_c in 10 mM SDS.

in the noncirculating range. The mass transfer coefficients for naphthalene in 50 mM SDS are shown in Fig. 7. All the k_c values calculated from experimental data appear to be best described by the correlations for noncirculating drops. The standard correlations for continuous-phase mass transfer coefficients for noncirculating drops appear to be applicable to design of extractors for SDS clean-up.

The presence of SDS does appear to reduce the mass transfer coefficient, perhaps by decreasing the hexane drop circulation as indicated in Figs. 5–7 and as suggested by Garner et al. (19), Thorsen and Terjesen (14), and Beitel and Heideger (21). A trade-off exists between the greater solubilizing power of high SDS concentrations and the greater mass transfer resistance to removal of pollutants from the more concentrated solu-



* noncirculating

Δ circulating

* & Δ circulating and noncirculating

FIG. 7 Comparison of experimentally determined k_c values to correlations for k_c in 50 mM SDS.

tions. The column experiments in which many hexane drops are passed through the contaminated SDS solutions at a time do, however, indicate that, at higher concentrations of SDS, contaminants can be removed fairly quickly, although the residual contaminant concentrations are higher than those observed when solutions having lower SDS concentrations are extracted.

CONCLUSIONS

The continuous-phase mass transfer coefficient was found to decrease with increasing SDS concentration, as shown in extraction studies with single drops of hexane. The presence of SDS was also found to decrease hexane drop rise velocities by about 30%. The hexane drops in the SDS

solutions reached a maximum velocity at a drop diameter of about 0.16 cm; above this the velocity decreased. The mass transfer coefficient also reached a maximum value corresponding to the same drop diameter. Correlations for continuous-phase mass transfer coefficients indicate that the k_c values in SDS correspond to those for noncirculating drops. Standard correlations for k_c for noncirculating drops may be used for design of extraction systems for reclaiming SDS solutions.

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