

Research Article

Oxygen Absorption into Stirred Emulsions of n-Alkanes

Thanh Hai Ngo and Adrian Schumpe

Institut für Technische Chemie, Technische Universität Braunschweig, Hans-Sommer-Straße 10, 38106 Braunschweig, Germany

Correspondence should be addressed to Thanh Hai Ngo, t-h.ngo@tu-bs.de

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Absorption of pure oxygen into aqueous emulsions of n-heptane, n-dodecane, and n-hexadecane, respectively, has been studied at 0 to 100% oil volume fraction in a stirred tank at the stirring speed of 1000 min^{-1} . The volumetric mass transfer coefficient, $k_L a$, was evaluated from the pressure decrease under isochoric and isothermal (298.2 K) conditions. The O/W emulsions of both n-dodecane and n-hexadecane show a $k_L a$ maximum at 1-2% oil fraction as reported in several previous studies. Much stronger effects never reported before were observed at high oil fractions. Particularly, all n-heptane emulsions showed higher mass-transfer coefficients than both of the pure phases. The increase is by up to a factor of 38 as compared to pure water at 50% n-heptane. The effect is tentatively interpreted by oil spreading on the bubble surface enabled by a high spreading coefficient. In W/O emulsions of n-heptane and n-dodecane $k_L a$ increases with the dispersed water volume fraction; the reason for this surprising trend is not clear.

1. Introduction

Oxygen absorption into emulsions is encountered, for example, in fermentations with an oil as the carbon source. Most studies have been carried out at low oil volume fractions typical for this application [1, 2]. Literature data on the effect of n-alkane addition on the volumetric mass transfer coefficient $k_L a$ for oxygen are illustrated in Figure 1. The $k_L a$ value has been reported to increase [3, 4], decrease [5, 6], or remain unaffected [7] compared to the one in water. Da Silva et al. [8] reported that 1% n-hexadecane or n-dodecane increased $k_L a$ in a stirred tank by factors of 1.68 and 1.36, respectively; Kundu et al. [3] found that addition of 1% n-dodecane or n-heptane could enhance oxygen transfer in a bubble column up to fourfold; Jia et al. [9] also found a fourfold increase by 2% soybean oil in an air-lift reactor. Among other factors, the oil spreading coefficient S (1) has been used to explain the differences between the oils as follows:

$$S = \sigma_{\text{WG}} - \sigma_{\text{OG}} - \sigma_{\text{OW}}. \quad (1)$$

The values of spreading coefficients S reported in the literature for the three n-alkanes used in this study, n-heptane, n-dodecane, and n-hexadecane, differ considerably (Table 1).

In the present work, absorption of pure oxygen into aqueous emulsions of these n-alkanes has been studied in the full range of oil volume fraction (0 to 100%) with a barometric technique. The main advantage of the pressure technique is that it can give information on both $k_L a$ and oxygen solubility. At high-oil volume fractions, not considered in the previous studies, the high oxygen solubilities in the oils (high driving force) should have a strong effect on the mass transfer characteristics.

2. Experimental

2.1. Chemicals. The oxygen gas had a purity of 99.999% (Westfalen AG, Germany). The emulsion (liquid phase) was composed of double-distilled water and n-heptane, n-dodecane, or n-hexadecane, respectively, with purities $\geq 99\%$ (Merck, Germany).

2.2. Spreading Coefficient (S). The spreading coefficient S was calculated based on the values of the surface and interfacial tensions measured at 298.2 K with a tensiometer (K11, Krüss, Germany) using the ring method [10]. The measurements were carried out with mutually saturated liquid phases. The reported surface tensions are mean values

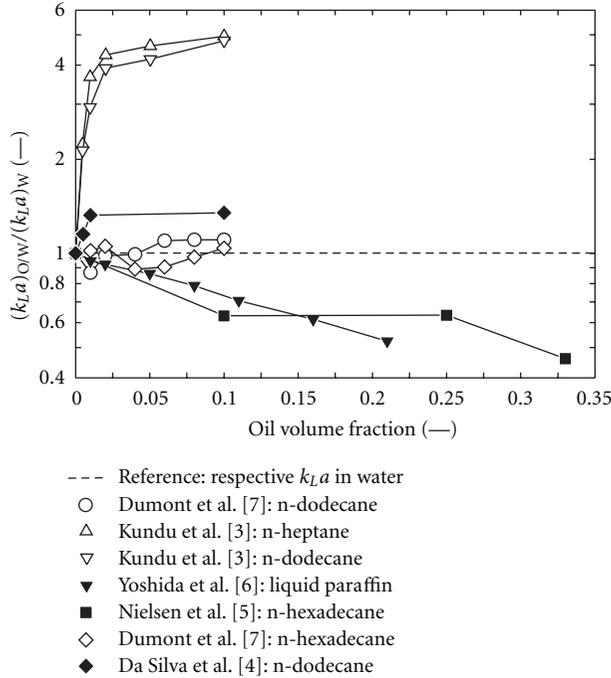


FIGURE 1: Variation of $k_L a$ for oxygen upon n-alkane addition reported in the literature for stirred tanks (filled symbols) and bubble columns (open symbols).

TABLE 1: Literature values of spreading coefficient S .

Oil	T (K)	S (mN m^{-1})	Authors
n-heptane	298.15	+1.2 to +2.3	Pinho and Alves [11]
	298.15	-5.3 to -5.9	Pinho and Alves [11]
	303.15	-2.6	Hassan and Robinson [12]
n-dodecane	298.15	+3.7	Oliveira et al. [13]
	?	+8.7	Wei and Liu [14]
	298.15	+0.6	Rols et al. [15]
n-hexadecane	303.15	-9.3	Hassan and Robinson [12]

of 3 measurements (maximum relative standard deviation 0.25%) and the interfacial tensions are mean values of 4 measurements (maximum relative standard deviation 4%). It should be noted that the aim was to understand the behavior of the oils used in this study; therefore, the oils were used without purification.

The evaluated spreading coefficients S are listed in Table 2. Different from the literature data (Table 1), all three oils have positive S values, but the value for n-heptane is noticeably higher than those for n-dodecane and n-hexadecane.

2.3. Volumetric Mass Transfer Coefficient ($k_L a$) and Oxygen Solubility. The mass transfer experiments were carried out in a stirred glass vessel with a total volume of 1830 mL and an inner diameter of 100 mm. A schematic diagram of the experimental setup is shown in Figure 2. The stirrer shaft was equipped with two pitched-blade PTFE stirrers (50 mm diameter, 4 blades) mounted at 20 mm (liquid phase) and 140 mm height (gas phase). Four baffles with a width of

TABLE 2: Measured values of the surface tensions of the mutually saturated water (σ_{WG}) and oil (σ_{OG}) phase, interfacial tension (σ_{OW}), and spreading coefficient S at 298.2 K [10].

Oil	σ_{WG}	σ_{OG}	σ_{OW}	S
	(mN m^{-1})			
n-heptane	71.8	19.8	40.2	11.8
n-dodecane	68.2	24.6	41.6	2.0
n-hexadecane	71.2	26.7	42.3	2.2

10 mm were mounted to the walls. The reactor was jacketed and the temperature was controlled to 298.2 ± 0.1 K with a thermostat. All experiments were carried out under surface aeration with gas entrainment into bubbles at the same high stirring speed (1000 min^{-1}).

The volumetric mass transfer coefficient ($k_L a$) was evaluated from the pressure decrease measured during batchwise saturation of the initially gas-free emulsion under isothermal and isochoric conditions. To this end, the desired mixture of oil and water (total volume 700 mL) was placed in the reactor and degassed by applying vacuum under stirring. The liquid losses during degassing were determined using a cold trap with liquid nitrogen as the coolant. About 1.3 mL n-heptane loss was compensated by adding the respective volume in excess, whereas the losses were negligible for n-dodecane and n-hexadecane.

After degassing, the stirrer was stopped, and the reactor was pressurized in about 10 s to approximately 0.15 MPa with pure oxygen. After about 20 s, when the pressure reading had become constant, the absorption experiment was started by switching on the agitator. The time course of the pressure decrease in the head space was measured with a pressure transducer ($0\text{--}0.5 \text{ MPa} \pm 0.05\%$). A typical experimental record is shown in Figure 3.

The volumetric mass transfer coefficient $k_L a$ was evaluated based on a pseudohomogenous model, that is, the oil-water emulsion was treated as a pseudohomogenous phase with mean physicochemical properties, specifically, mean gas solubility. This allowed determining the $k_L a$ -value from the decrease of pressure P from initially P_i to finally P_f (see Figure 2) using (2) suggested by Albal et al. [16]:

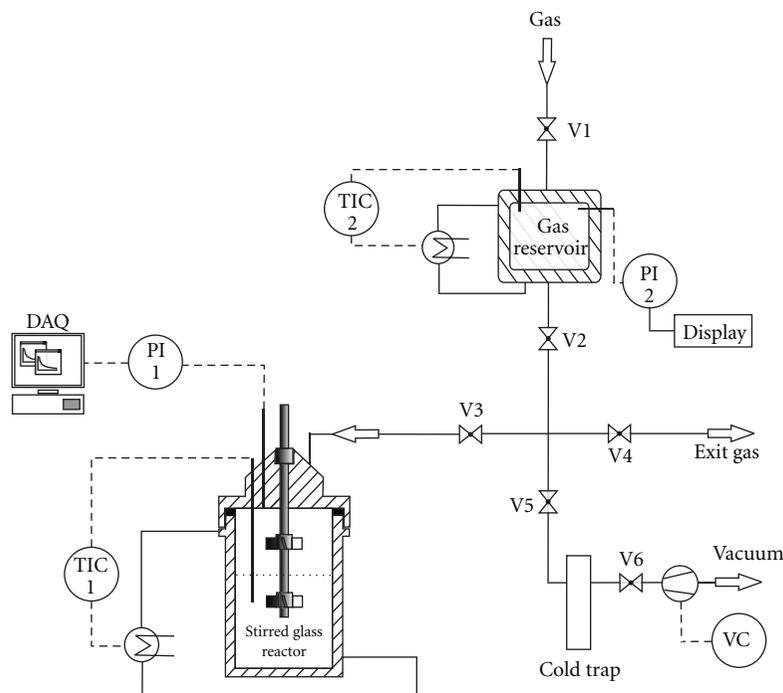
$$-\frac{P_f - p_W - p_O}{P_i - p_W - p_O} \cdot \ln(P - P_f) = k_L a \cdot t + \text{const.} \quad (2)$$

Here p_W and p_O are the partial pressures of water and oil, respectively. The data at 15% to 90% saturation was used to evaluate $k_L a$ in order to exclude the initial re-emulsification phase as well as the final phase with low driving force. The reported $k_L a$ values are mean values of at least 5 measurements; the mean relative standard deviation was 8% for n-dodecane and n-heptane emulsions, and 7% for n-hexadecane emulsions.

From the total pressure decrease, ΔP , the overall oxygen solubility in the emulsion at a partial pressure of 101325 Pa can be calculated as follows:

$$C_{O_2}^* = \frac{V_G}{V_L} \cdot \frac{1}{RT} \cdot \Delta P \cdot \frac{101325 \text{ Pa}}{P_f - p_W - p_O}. \quad (3)$$

For the oxygen solubility, the mean relative standard deviation was always less than 5%.



DAQ: acquisition computer
 PI: pressure sensor
 TIC: temperature controller
 VC: vacuum controller

FIGURE 2: Experimental setup.

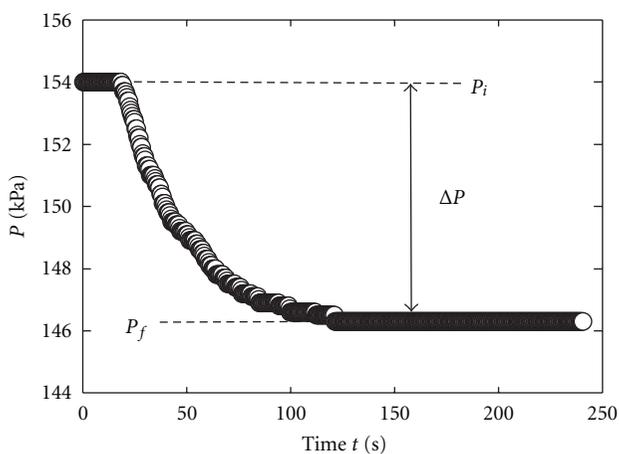


FIGURE 3: Pressure decrease during O₂ absorption into n-dodecane emulsion ($\Phi_{oil} = 30\%$).

3. Results and Discussion

3.1. Effect of Oil Volume Fraction on O₂ Solubility. The oxygen solubilities in the emulsions increases linearly with oil volume fraction (Figure 4). The oxygen solubility decreases with increasing chain length of the n-alkanes. The relative

solubility (oxygen solubility in the pure oil compared to pure water) is 11.1, 7.7, and 6.5 for n-heptane, n-dodecane, and n-hexadecane, respectively.

3.2. Phase Inversion. In the previous study on CO₂ absorption [10], the electrical conductivity method showed the phase inversion taking place in the range of 60% to 65% oil volume fraction for n-dodecane as well as n-hexadecane. For n-heptane, the conductivity variation was rather continuous; observation of deemulsification suggested the inversion region to be at 50%–60% n-heptane.

3.3. Effect of Oil Volume Fraction on $k_L a$. The volumetric mass transfer coefficient $k_L a$ in O/W emulsions of n-dodecane and n-hexadecane show similar trends (Figures 5 and 6): As the oil volume fraction increases, $k_L a$ first increases to a maximum at a small oil volume fraction and then decreases towards the phase inversion region. The increase in $k_L a$ at 1% n-dodecane and at 2% n-hexadecane is by 44% and 26%, respectively. Note that the standard deviation, indicated by error bars, is higher in this range. An increase in $k_L a$ at low oil concentrations (1-2%) was previously observed for oxygen by several authors. According to Clarke and Correia [1] hydrocarbons tend to decrease

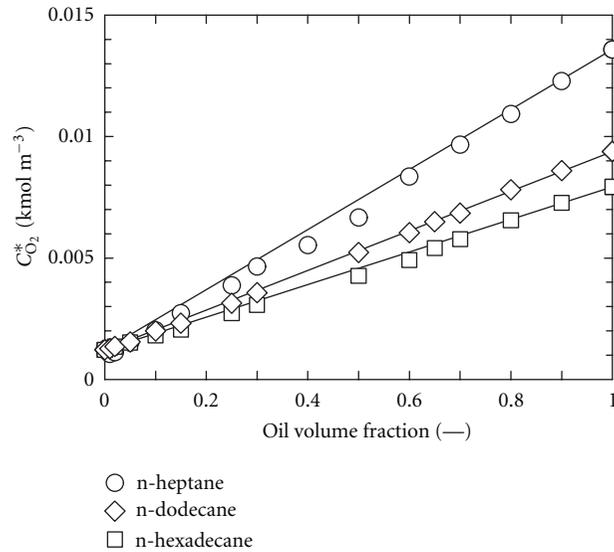


FIGURE 4: Effect of oil volume fraction on O_2 solubility at a partial pressure of 101325 Pa.

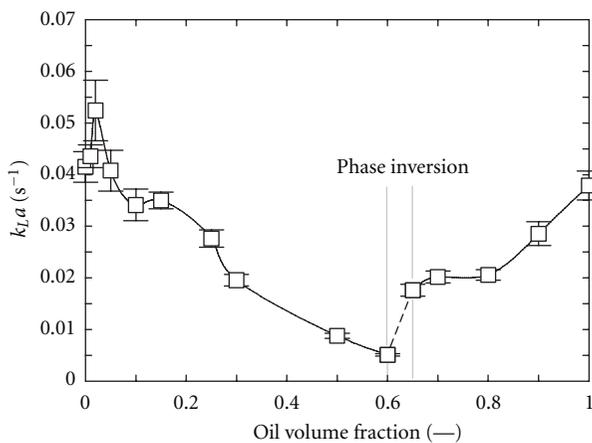


FIGURE 5: Effect of n-hexadecane volume fraction on $k_L a$.

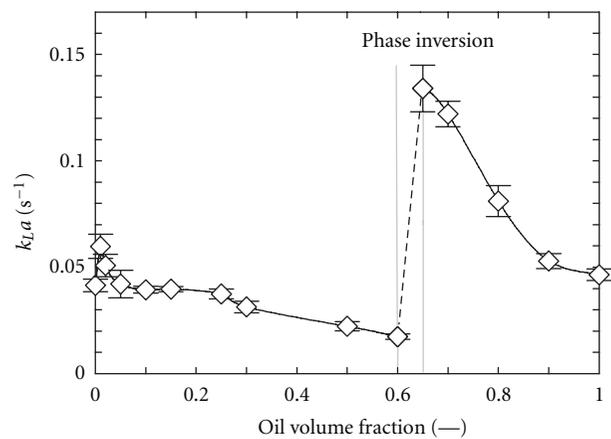


FIGURE 6: Effect of n-dodecane volume fraction on $k_L a$.

the surface tension of the water phase, resulting in smaller bubble size, therefore higher interfacial area (a), and hence higher $k_L a$. However, in this study the surface tension of water (72.0 mN m^{-1}) and that of water saturated with n-heptane (71.8 mN m^{-1}) or n-hexadecane (71.2 mN m^{-1}) were practically the same (cf. Table 2); only saturation with n-dodecane slightly reduced the surface tension (68.2 mN m^{-1}). This means that the “surface tension effect” is negligible for the systems studied. Bruining et al. [17] and Cents et al. [18] proposed that an additional transport mechanism might be responsible for the maximum at low oil fraction. Small oil droplets with higher solubility for the gas might enter the water film at the G/L interface, absorb the gas, and then return to the bulk water phase where the absorbed gas is discharged. After the maximum, the strong decrease of $k_L a$ is quite expected due to the increase of the emulsion viscosity. All trends are similar to those for CO_2 absorption in the same emulsions [10] but the effects are much more pronounced for oxygen. The main difference between the gases is that the

CO_2 solubilities in the organic liquids are only slightly higher than in water.

The O/W emulsions of n-heptane show quite different trends with increasing oil fraction (Figure 7): $k_L a$ strongly increases up to 15% oil fraction and then again from 40% oil towards the phase inversion region. All n-heptane emulsions have higher volumetric mass transfer coefficients than pure water.

The increase in $k_L a$ at 15% n-heptane volume fraction (+336% as compared to water) can probably be explained by the bubble-covering mechanism proposed by Rols et al. [19]. This mechanism is linked to the spreading coefficient S . Pinho and Alves [11] recently concluded that there is direct contact between gas and oil when $S > 0$, and very high mass transfer coefficients can be expected. The high S value for n-heptane (Table 2) might enable n-heptane to spread as a film on the oxygen bubbles. When a bubble finally breaks at the top surface, small oxygen-rich droplets may leave the oil film and release oxygen to the water phase. It should be

noted that n-heptane has the highest oxygen solubility of the three alkanes used and the $k_L a$ value in pure n-heptane is remarkably higher than that in pure water (+358%), whereas the $k_L a$ values in pure n-dodecane and in pure n-hexadecane are similar to the one in pure water. This means that if n-dodecane or n-hexadecane could also spread as a thin film on the oxygen bubbles, the mechanism would be less effective. The differences of the $k_L a$ values in the pure oils mainly result from the viscosities [20] of only 0.39 mPa s for n-heptane and 1.38 mPa s and 3.03 mPa s for n-dodecane and n-hexadecane, respectively.

In W/O emulsions, from 100% n-hexadecane down to the phase inversion region, $k_L a$ monotonously decreases with increasing dispersed water fraction. This trend is expected due to the increase in viscosity of the W/O emulsions with increasing water content [21]. Very surprisingly, for both n-heptane and n-dodecane, from 100% oil volume fraction down to the phase inversion region, $k_L a$ does not decrease but increases substantially! The maximum increase in $k_L a$ compared to the pure oil is found at the phase inversion region where it is +101% for n-heptane and even +189% for n-dodecane. (For n-dodecane the $k_L a$ jump at the phase-inversion point is by a factor of 7.8). More extensive repetitions than usual have verified these results. To find an explanation, the structure of the W/O emulsion was studied with an endoscopic photographic technique [10, 22, 23]. The endoscopic photoprobe was installed at about half the emulsion height; after about 2.5 min. of stirring at 1000 rpm, photos were taken at 10 fps (frames per second). Figure 8 is a typical photo taken at 70% n-dodecane volume fraction. In this W/O emulsion, the water drops have a surface-to-volume mean diameter of 1.16 mm (standard deviation: 0.15 mm; count: 1142 droplets). There are some very tiny droplets but there is no multiemulsion (e.g., O/W/O emulsion).

It may be argued that the driving force should be based on the solubility $(C_{O_2}^*)_{\text{cont}}$ in the continuous phase rather than the mean solubility as assumed in (2). This will shift the $k_L a$ values in O/W emulsions upwards (lower driving force) while those in W/O emulsions will be shifted downwards (higher driving force). Nielsen et al. [5] additionally suggested a comparison with the volumetric mass transfer coefficient in the respective continuous phase $(k_L a)_{\text{cont}}$ in the form of the mass transfer enhancement factor E as follows:

$$E = \frac{k_L a}{(k_L a)_{\text{cont}}} \cdot \frac{C_{O_2}^*}{(C_{O_2}^*)_{\text{cont}}} \quad (4)$$

One might expect E to decrease from 1 on both sides to lower values at the phase inversion point due to the viscosity effect. Figures 9 and 10 present the effects of oil volume fraction on the mass transfer enhancement factor E . In O/W emulsions of both n-dodecane and n-hexadecane, as the oil fraction increases, at 25% oil fraction maxima are observed. There the mass transfer rate is enhanced by a factor of 2.3 for n-dodecane and a factor of 1.5 for n-hexadecane.

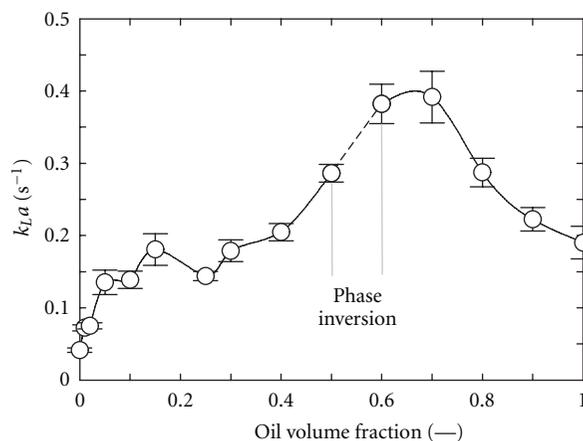


FIGURE 7: Effect of n-heptane volume fraction on $k_L a$.

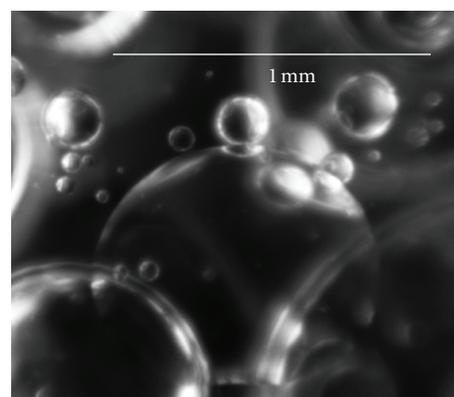


FIGURE 8: Photo of W/O emulsion at 70% n-dodecane taken with the SOPAT endoscope (SOPATec, Berlin, Germany).

At further increase in the oil fraction towards the phase-inversion region, the E value for n-dodecane decreases but still shows some mass transfer enhancement, whereas mass transfer retardation is observed above 40% n-hexadecane. This difference reflects the stronger $k_L a$ decrease in the case of n-hexadecane (Figure 5) as compared to n-dodecane (Figure 6), for example, at 60% oil fraction $k_L a$ is lower than in water by a factor of 0.12 for n-hexadecane and for n-dodecane the factor is only 0.42. In addition, the relative oxygen solubility in n-hexadecane as compared to water is 6.5 whereas it is 7.7 for n-dodecane. Both aspects lead to higher E values for n-dodecane (Figure 8).

In the case of O/W emulsions of n-heptane (Figure 10), as the oil volume fraction is increased towards the phase-inversion region, high mass transfer enhancement is always observed. This reflects the high $k_L a$ values (Figure 7) as well as the high relative oxygen solubility of 11.1 in n-heptane as compared to water. Mass transfer is enhanced by $E = 37.7$ at the heptane volume fraction of 50%. The trend in O/W emulsions might be explained by the bubble-covering mechanism. However, further research is needed to explain the surprising increase in $k_L a$ and E with increasing water

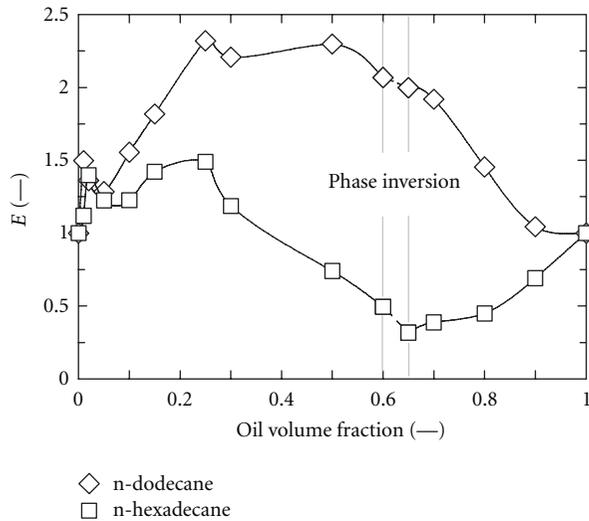


FIGURE 9: Variation of the mass transfer enhancement factor E with the n-dodecane and the n-hexadecane volume fraction.

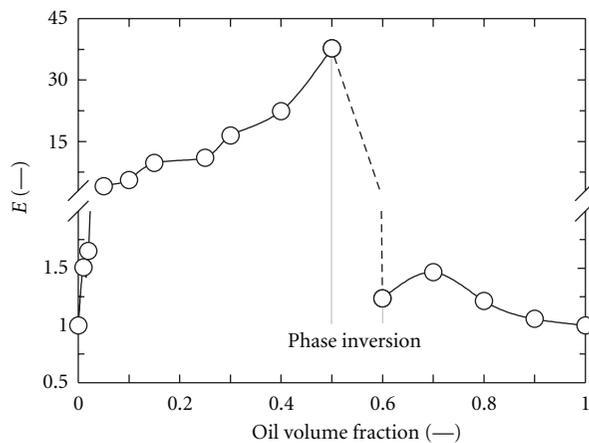


FIGURE 10: Variation of the mass transfer enhancement factor E with the n-heptane volume fraction.

content (from 100% oil down to the phase inversion region) for W/O emulsions of n-dodecane and n-heptane.

4. Conclusions

Oxygen absorption into aqueous emulsions of n-heptane, n-dodecane, and n-hexadecane has been studied in the full range of oil volume fraction. The oxygen solubility in emulsions of all three n-alkanes increases linearly with the oil volume fraction. All three oils have positive spreading coefficients S but a particularly high S value is found for n-heptane. In O/W emulsions, $k_L a$ maxima at low oil fraction (1-2%) are found for n-dodecane and n-hexadecane. Emulsions of n-heptane always show higher $k_L a$ than the pure liquids. This could be explained by a bubble-covering mechanism enabled by high spreading coefficient. In W/O emulsions of both n-heptane and n-dodecane, $k_L a$ increases with increasing dispersed water content towards a maximum

at the phase-inversion region. This is a quite unexpected trend that still needs to be explained.

Nomenclature

a :	Specific interfacial area [m^{-1}]
$C_{O_2}^*$:	Oxygen solubility [kmol m^{-3}]
$k_L a$:	Volumetric mass transfer coefficient [s^{-1}]
k_L :	Mass transfer coefficient [m s^{-1}]
$llP_i(P_f)$:	Initial (final) total pressure [Pa]
$p_O(p_W)$:	Partial pressure of oil (water) [Pa]
R :	Gas constant [$\text{Pa m}^3 \text{kmol}^{-1} \text{K}^{-1}$]
S :	Spreading coefficient [N m^{-1}]
T :	Temperature [K]
t :	Time [s]
V_G :	Gas phase volume [m^3]
V_L :	Emulsion volume [m^3]
$\sigma_{WG}(\sigma_{OG})$:	Surface tension of water (oil) [N m^{-1}]
σ_{OW} :	Oil-water interfacial tension
Φ_{oil} :	Oil volume fraction [—]
E :	Mass transfer enhancement factor [—].

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