

as inferred from Sridharan and Sharma (1976) and Alvarez-Fuster et al (1980). The effective concentration driving force was thus based upon the CO₂ concentration relative to the equilibrium value. However, equilibrium was never reached over the course of any experiment. The ratio of equilibrium driving force ($[\text{CO}_2^*] - [\text{CO}_2^*]_{\text{eq}}$) to concentration driving force ($[\text{CO}_2^*]$) exceeded 0.85 in all experiments. The presence of a reaction equilibrium thus only had a minor effect upon the rate of transfer of CO₂. To a first approximation, a simplified rate expression could be used:

$$R_{\text{r}}a = a[\text{CO}_2^*][\text{CHA}_0]\sqrt{D_{\text{CO}_2}k_{12}} \quad (9)$$

The complete expression was retained in calculations for accuracy, however.

The value of the recycle ratio, R , could not be determined independently in this study. Since the conversion of CO₂ was often as great as 80% initially, the calculated value of a was very dependent upon the postulated residence time gas distribution as characterized by R . Thus a as calculated could vary by as much as five-fold with the extreme limits of the value of R . Consequently, only relative values of a are reported here. These were determined by assuming that the gas phase RTD remained constant at a fixed stirring speed as solids loading was changed. In this study $R = 0$ (PFR) was used as the basis for comparison of the interfacial areas.

The discrepancy among interfacial areas caused by uncertainty in the gas phase RTD has been addressed by Schumpe and Deckwer (1980) and Midoux et al (1980). They compared the value of a calculated for a particular bubble size distribution with the value of a calculated for a simplification of the gas phase RTD (either PFR [a_{PFR}] or CSTR [a_{CSTR}]). What seems clear is that the values of a calculated for all possible choices of bubble size distributions converge when the calculated values of a_{PFR} and a_{CSTR}

converge. This criterion can be used to establish when a particular reaction is suitable for calculating a independent of any gas phase assumptions.

The simplification for R_f (Equation 9) can be substituted into Equation 4 together with Equations 6 and 7 to yield an integrated expression for x_0 :

$$St_1 = \frac{ay_i H^* P \sqrt{D_{CO_2} k_{12} [CHA_o]^2}}{\left[\frac{Cy_i}{V} \right]} = x_i + x_o + (R + 1) \log_e \left[\frac{x_o R + x_i}{x_o (R + 1)} \right] \quad (10)$$

A dimensionless mass Stanton number can be used to characterize the system. As formulated above, St_1 is the ratio of the amount of CO_2 transferred into the liquid to the total amount present available for transfer. At low St_1 values the solutions to this equation in x_0 are indistinguishable for varying values of R . Ranges in St_1 for typical reactions used in determining a are indicated in Table 4 and Figure 3. The physico-chemical constants of these reactions satisfy the criteria of Table 1. Figure 3 indicates why the sulfite method remains in use despite these new classes of reactions available for determining a : a can be determined without any a priori knowledge of the gas phase RID.

An attempt was made to lower the reaction rate constant for the CHA carbamation reaction by measuring interfacial areas at 5 °C instead of at 20 °C for which kinetic data was available. An estimated four-fold reduction in rate constant was effected, based upon the activation energy for similar carbamations (Alvarez-Fuster et al., 1980). Conversion of CO_2 exceeded 80% in all of these experiments, however, still requiring accurate knowledge of the gas phase RID to calculate a . This result can be

Table 4

Comparison of Different Chemical Systems
Used to Determine Interfacial Area

	$\text{CO}_2 + 2\text{CHA} \rightarrow$ <u>CHA.CO₂.CHA</u>	$\text{CO}_2 + 2\text{DEA} \rightarrow$ <u>DEA.CO₂.DEA</u>	$\text{O}_2 + 2\text{SO}_3^{-2} \rightarrow$ <u>2SO₄^{-2}</u>
Reference	1, 2	2	3
Solvent	90% toluene 10% isopropanol	ethanol	water
m	1	1	1
n	2	2	1
H* (kg.mol/m ³ .MPa)	1.0417	0.3788	0.0118
D _A (m ² /s)	4 X 10 ⁻⁹	1.9 X 10 ⁻⁹	2.4 X 10 ⁻⁹
k _{1n} (m ³ⁿ /kg.mol ⁿ .s)	2900	840	65-1628 (a)
[B ₀] (kg.mol/m ³)	0.25	0.25	0.80
H*√D _A k _{1n} [B ₀] ⁿ	89 X 10 ⁻⁶	12 X 10 ⁻⁶	0.2-4 X 10 ⁻⁶
VP/G (m ³ .s.MPa/mol)	280	280	280
a (m ⁻¹)	120-900	120-900	120-900
St ₁ (-)	3-23	0.4-3	0.006-0.5

$$St_1 = \frac{aVPH^* \sqrt{D_A k_{1n} [B_0]^n}}{C}$$

(a) k₁₁ for the sulfite oxidation is k₁₁' [Co⁺²] and thus varies with [Co⁺²]

References

- 1 Alvarez-Fuster et al. (1980)
- 2 Sricharan and Sharma (1976)
- 3 de Waal and Okeson (1966)

CHA = cyclohexylamine
DEA = diethanolamine
CHA.CO₂.CHA = carbamate of CHA
DEA.CO₂.DEA = carbamate of DEA

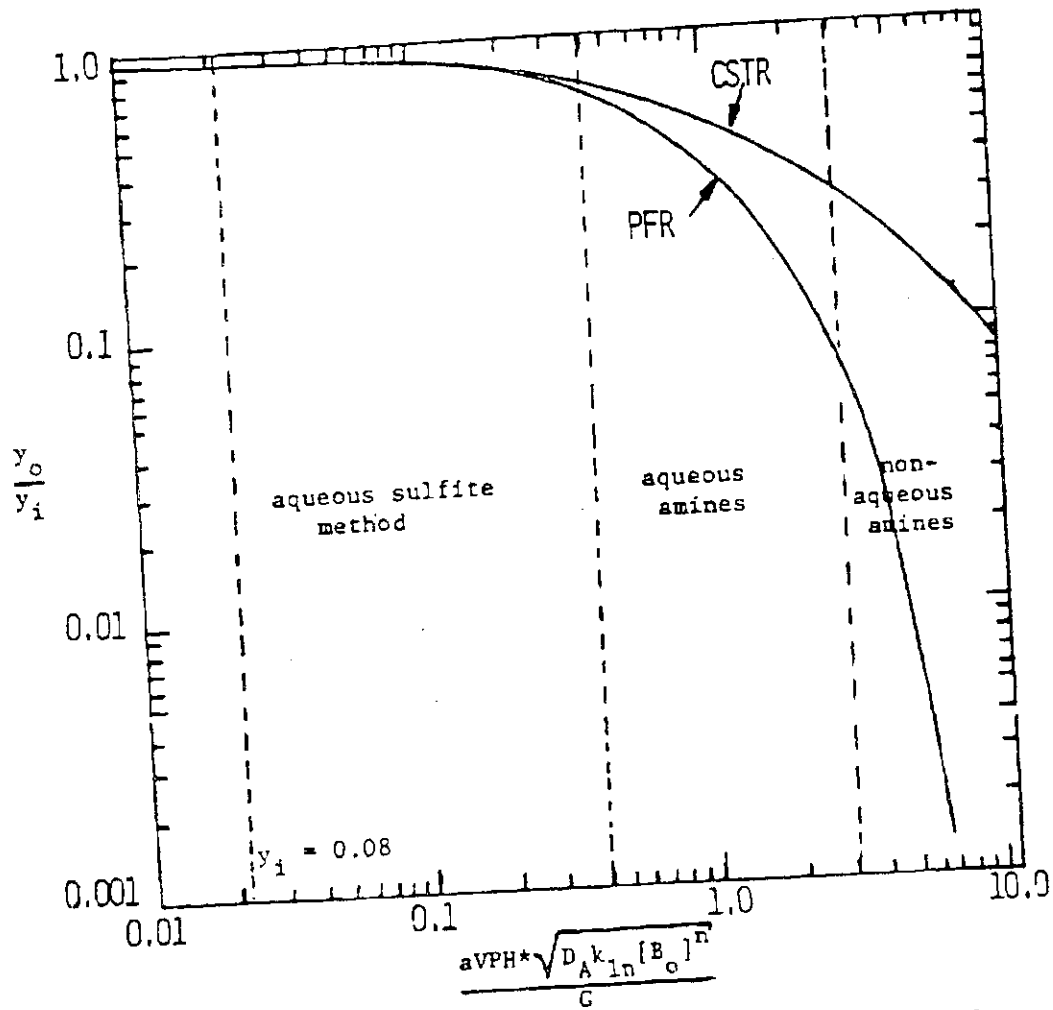


Figure 3. The convergence of the calculated interfacial area for varying gas phase assumptions is dependent upon the chosen reaction.

predicted by Figure 3, wherein a four-fold rate constant reduction translates to only a two-fold decrease in St_1 . This decrease is insufficient to be within the range where a_{PFR} and a_{CSTR} converge.

The overall mass transfer coefficient, $k_L a$, was calculated from a mass balance upon helium in the liquid and in the gas phase. The pertinent equations are as follows:

$$\text{gas:} \quad \epsilon_g V \frac{dC_{g,\text{ave}}^*}{dt} = k_L a V (C_L - C_i) - Q C_{g,o} \quad (11)$$

$$\text{liquid:} \quad V(1 - \epsilon_s) \frac{dC_L}{dt} = -k_L a V (C_L - C_i) \quad (12)$$

$$C_i = C_{g,\text{ave}}^* H \quad (13)$$

$$C_{g,\text{ave}}^* = C_{g,o} \left(\frac{R + 1/2}{R + 1} \right) \quad (14)$$

$$C_g = 0 \quad \text{at} \quad t = 0 \quad (15)$$

$$C_L = C_{L,\text{sat}} \quad \text{at} \quad t = 0 \quad (16)$$

The concentration of helium in the reactor, C_g^* , was modelled as a linear increase from the inlet to outlet value. The average concentration, $C_{g,\text{ave}}^*$, was thus the integrated average over the length of the reactor.

The resulting coupled differential equations yielded a solution for the outlet helium mole fraction which could be approximated by a decaying exponential at long times. The logarithm was thus taken of the outlet helium concentration, the slope of the linear region being approximately equal to $k_L a / (1 - \epsilon_s)$. This measurement was relatively insensitive to R . Variation of only 10% between $R = 0$ and $R = \infty$ was calculated for any given $k_L a$ measurement. Values of $k_L a$ could thus be determined independent of any

gas phase assumption.

In the Fischer-Tropsch studies, $k_L a$ was not measured directly, but was rather related to conversion (X). The method of analysis used was from Satterfield and Huff (1982), which related $k_L a$ and X as follows:

$$\frac{H_H^* p_H}{X S_V} = \frac{1}{k_{FT}} + \frac{7(1 - \epsilon_G)}{19k_{L,H} a} \quad (17)$$

A change in $k_L a$ thus did not result in a proportional change in conversion.

Results: Carbamation Studies

Experiments were first conducted without inert solids present to establish the variation of a and $k_L a$ with stirring speed. (Figures 4 and 5). Both varied linearly with stirring speed over the range encountered in this study, assuming $R = 0$. This linear relationship has also been reported by Mehta and Sharma (1971) and Sridhar and Potter (1982). When a CSIR in the gas phase was assumed ($R = \infty$), the calculated interfacial areas varied nonlinearly, showing sharper increases at higher stirring speeds. Various volume fractions of the solids indicated in Table 2 were then added to the systems at constant slurry volume to determine their effects upon a and $k_L a$ (Figures 6 and 7). A linear decrease in both a and $k_L a$ with volume fraction of inert solids was observed at all stirring speeds encountered in this study.

The decrease in a and $k_L a$ was independent of particle size over the range of sizes examined in this study. This can be observed by comparing the values of a and $k_L a$ for soda-lime glass in Figures 6 and 7. This result was also demonstrated by Ledakowicz et al. (1983), but only for $k_L a$. The decrease in both a and $k_L a$ was also independent of particle density. The hollow glass spheres used in this study were compositionally similar to

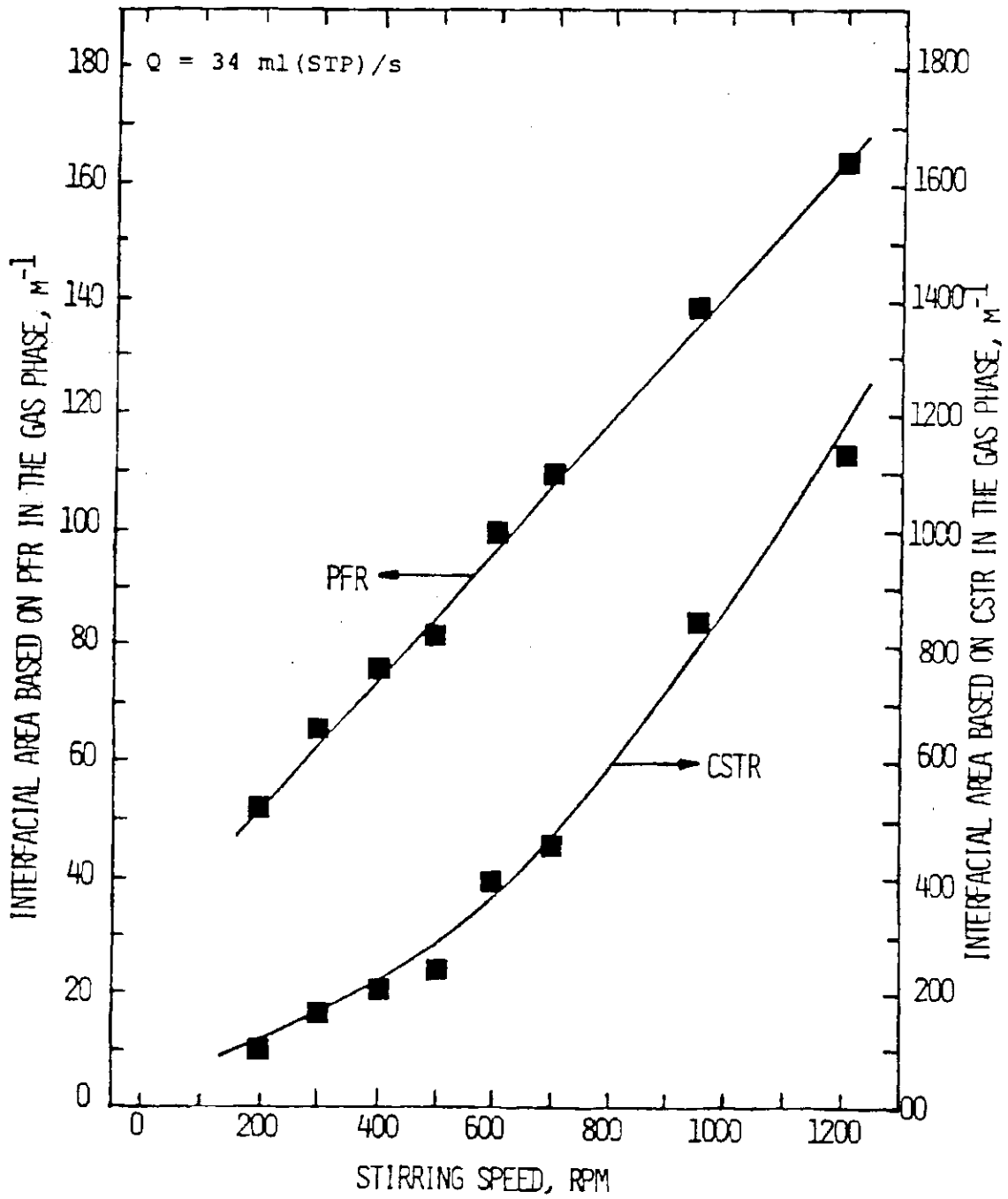


Figure 4. Interfacial area varies linearly with stirring speed at low values of R .

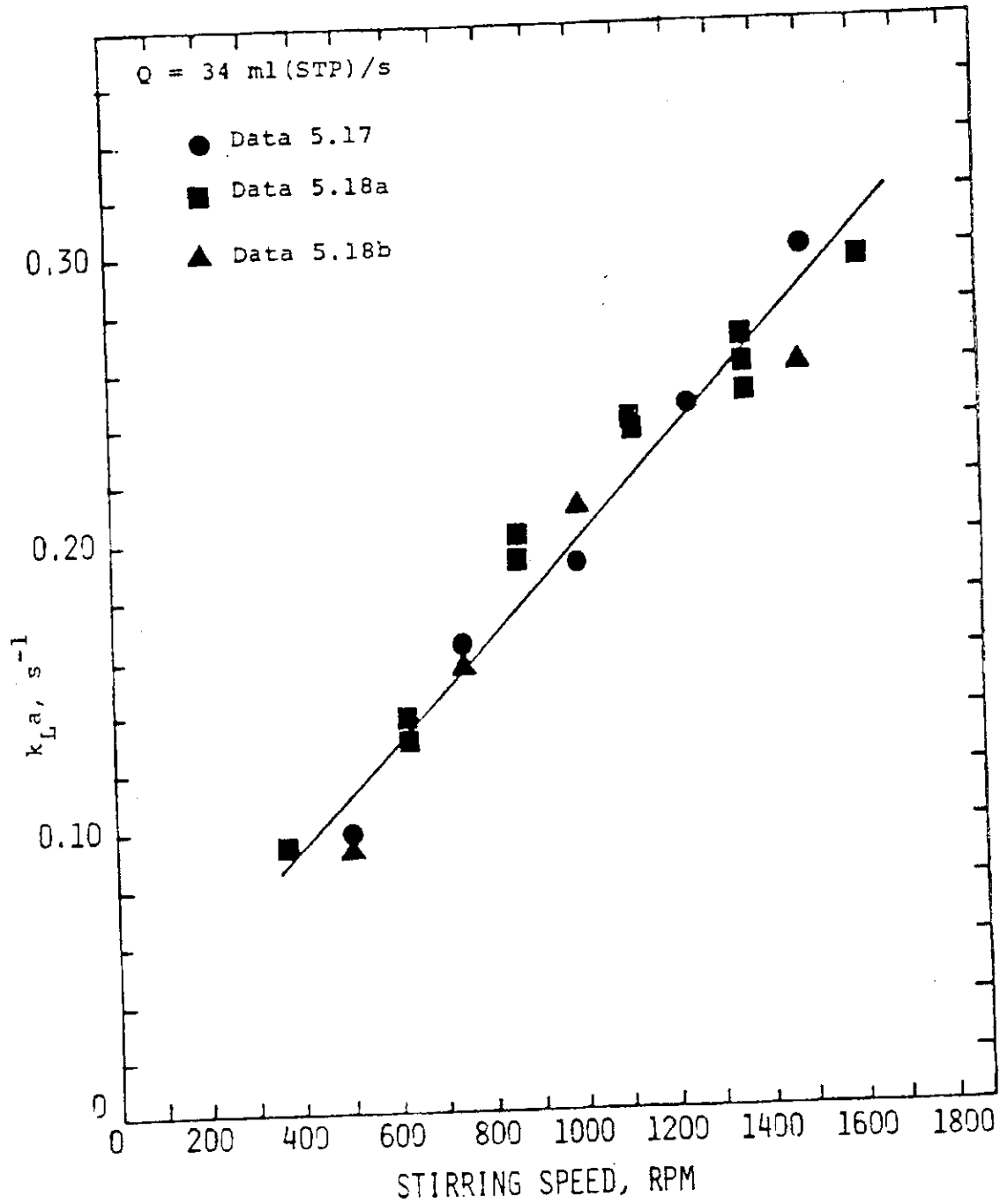


Figure 5. Overall mass transfer coefficient varies linearly with stirring speed.

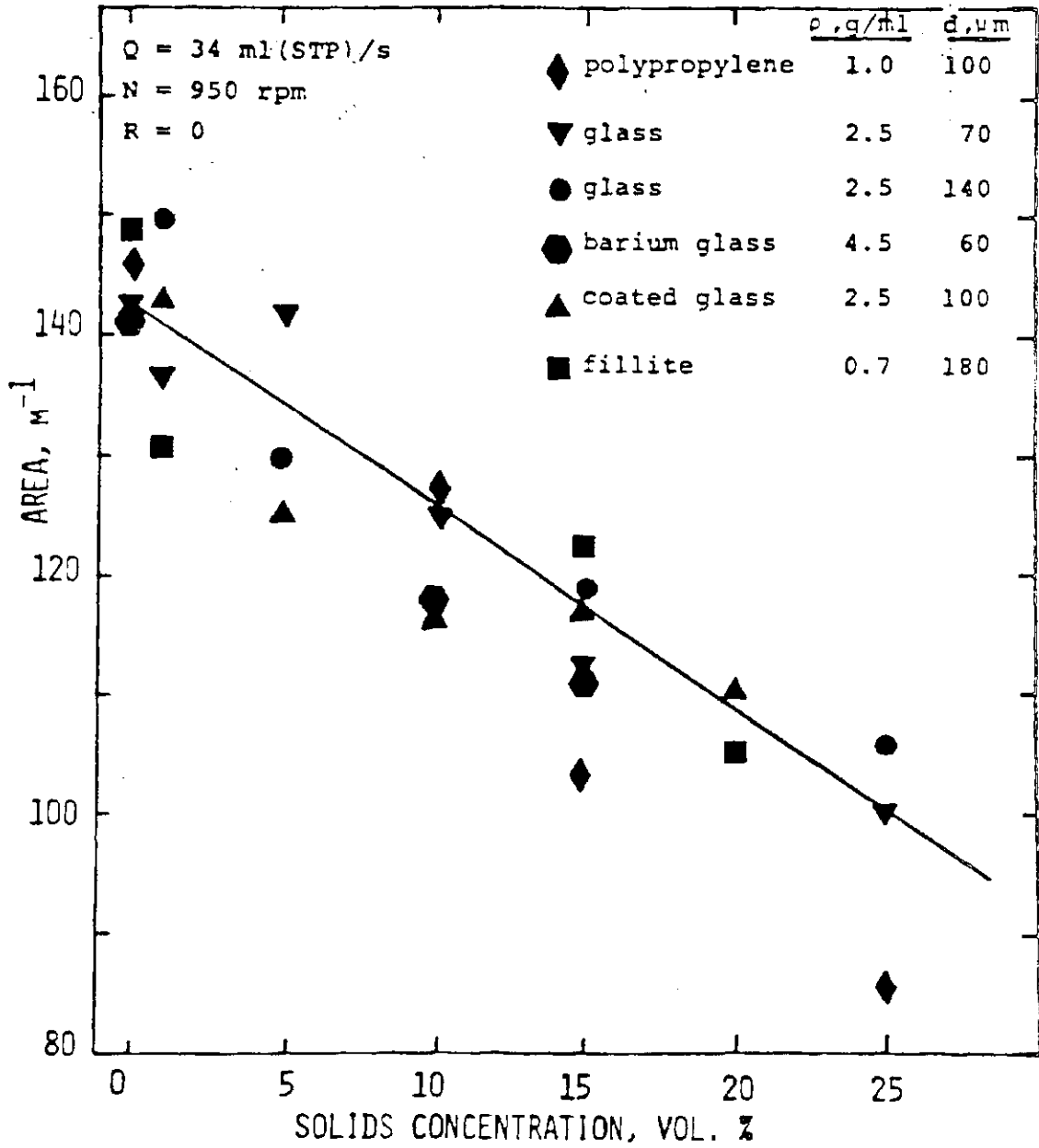


Figure 6. Interfacial area decreases with increased solids concentration.

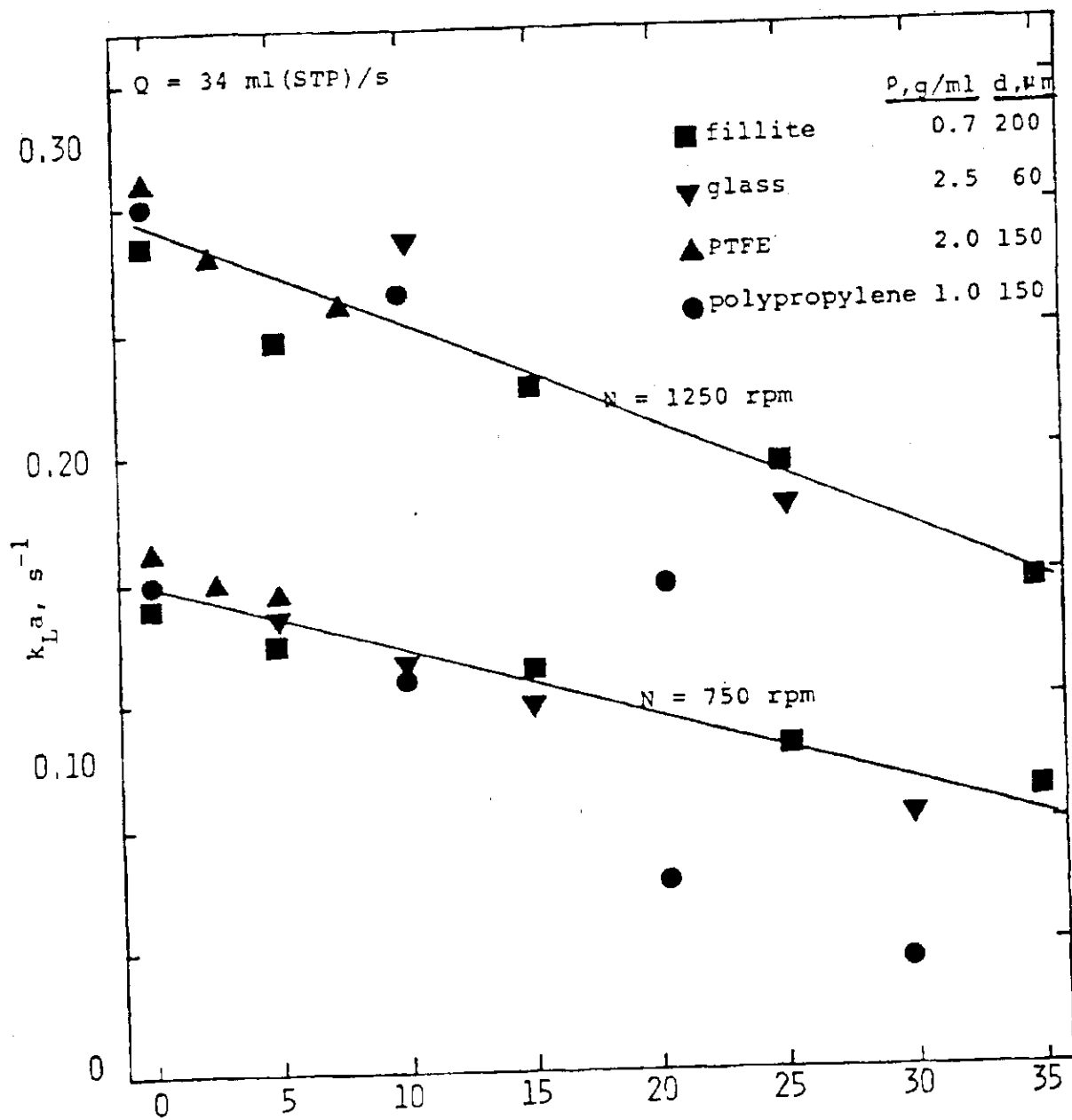


Figure 7. The overall mass transfer coefficient is dependent upon particle composition at high volume fractions of solids.

the solid glass beads, but varied in density by a factor of almost four. The effect of particle density was thus able to be tested free from the effects of particle composition. This independence of both a and $k_L a$ from particle density also indicates that stirring speed is the proper basis for comparison among gas-liquid-solid systems in mechanically stirred vessels. Constant power is often used as the basis for comparison in stirred vessels (Joosten et al., 1977); however, in this study the plots of $k_L a$ and a versus volume fraction solids would not have remained colinear for varying particle density had they been reduced to a constant power basis.

Results: Fischer-Tropsch Reaction Studies

The steady state measurements in the Fischer-Tropsch reactor showed no significant effect of inert particle loading upon conversion under partial mass-transfer limitation at 320 rpm (Figure 8). This result was obtained in both Fischer-Tropsch runs, which duplicated each other in most respects except for the choice of inlet H_2/CO ratio. Only the results of the second run are displayed here. These results seemingly do not parallel those obtained for the unsteady state $k_L a$ studies, which showed a decrease in $k_L a$ with solids loading. However, the mass transfer resistance in the Fischer-Tropsch reaction represented only about 20% of the total resistance to mass transfer. From the unsteady state determination of $k_L a$ the addition of up to 20 volume % of inert solids can cause a decrease in $k_L a$ of about 13% (Figure 7). When the slurry volume increase due to solids addition is accounted for in the Fischer-Tropsch reactor, the expected $k_L a$ decrease is about 10%. The predicted decrease in conversion in the Fischer-Tropsch reactor with inert solids addition based upon the unsteady state measurements of $k_L a$ was thus only calculated to be a maximum of about 2% (20% X 10%). The calculated and observed change in conversion matched within the

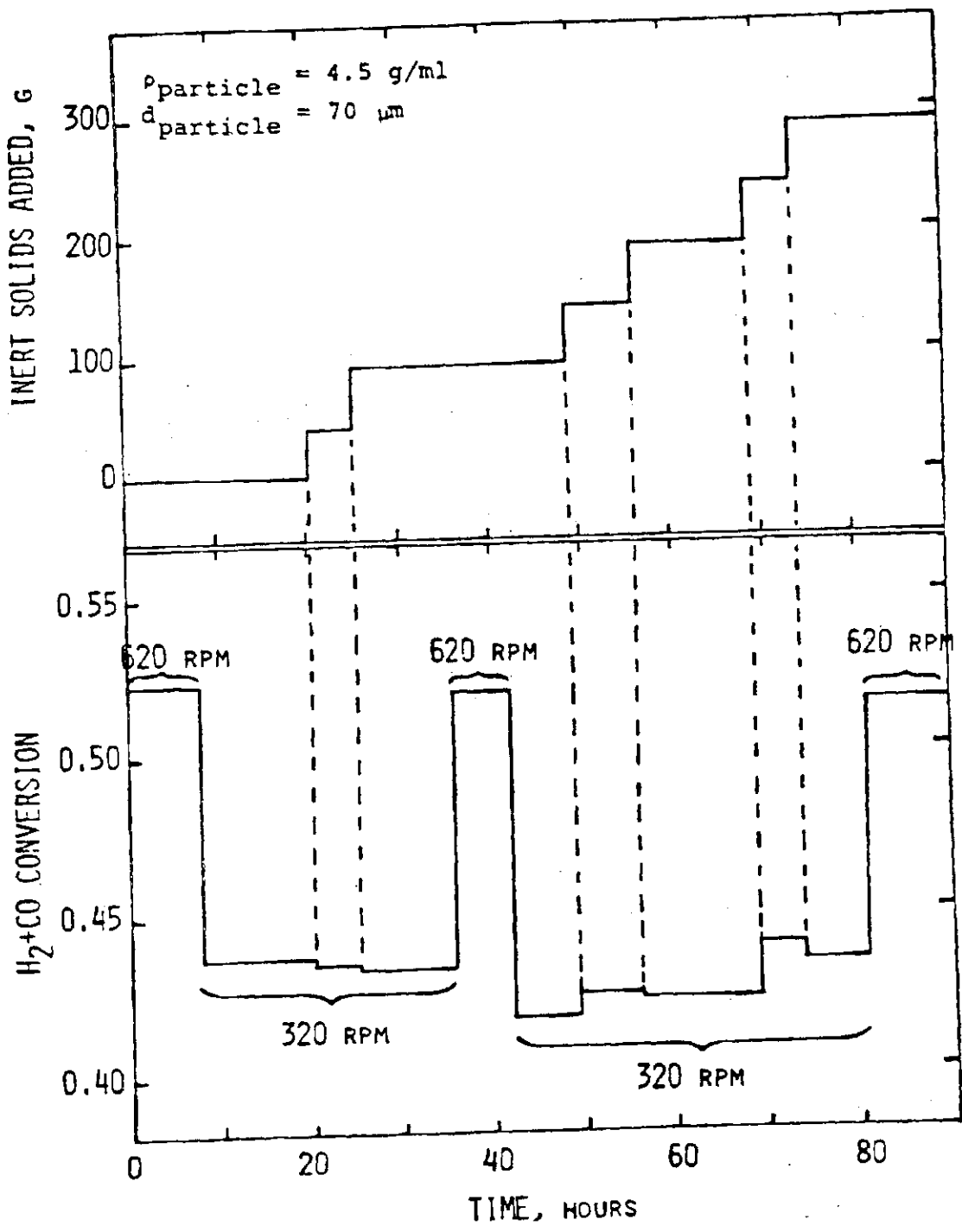


Figure 8. Conversion does not vary significantly with inert solids addition for Fischer-Tropsch synthesis under partial mass-transfer limitations.

accuracy of the measurement.

The amount of decrease of a and $k_L a$ with increased solids loading also varied moderately with particle composition. At high solids loadings, $k_L a$ and a decreased more in the presence of polypropylene than any of the glasses, even though the density of the polypropylene is between the extremes of the glasses. The polypropylene particles are granular, while the glasses are spherical, but this possible effect is discounted since the decrease in $k_L a$ is independent of particle size. This effect was also reported by Joosten et al. (1977) for $k_L a$ measurements, although attributed to density effects in their work.

Discussion

Measurements were performed to determine $k_L a$ and a in identical vessels using identical slurries. This procedure should thus allow direct calculation of k_L , based upon the ratio of $k_L a$ and a . However, imprecise knowledge of the gas phase RID did not allow a to be calculated exactly. The values indicated in the figures are based upon $R = 0$. If R is assumed constant at a fixed stirring speed, though, the relative effect of particle loading upon k_L can be ascertained. Figure 9 indicates the variation in k_L with solids loading at 950 rpm. This is the value of k_L for helium, since the $k_L a$ experiments were conducted with helium desorption. The value of a in the absorption with reaction experiment was assumed the same as the value of a in the $k_L a$ measurement. Sharma and Mashelkar (1968) demonstrated that a was independent of gas composition, justifying this assumption.

No variation in k_L with solids loading was observed, indicating that the decrease in $k_L a$ can be attributed entirely to a decrease in a . This result is consistent with the work of Calderbank and Moo-Young (1961).

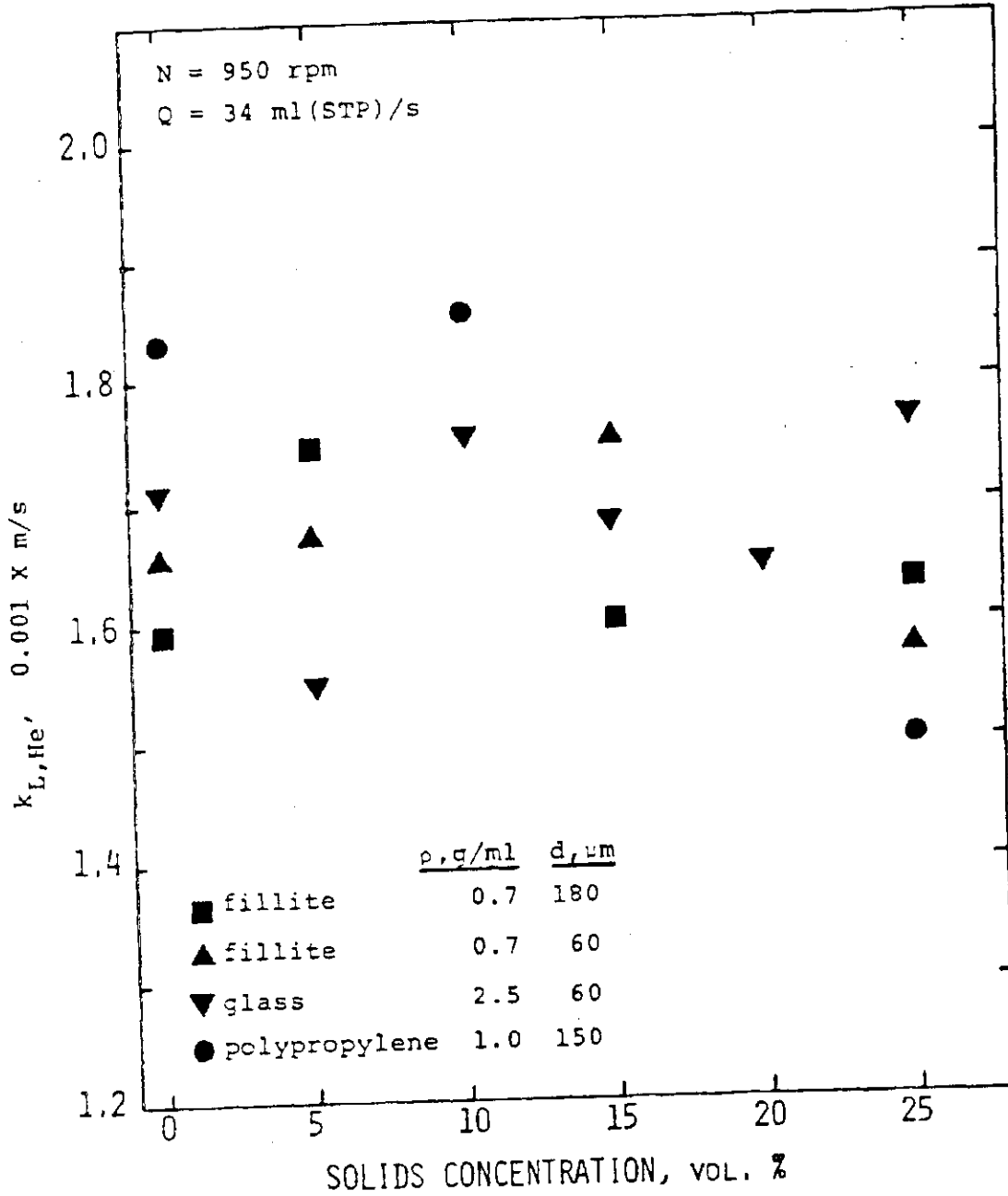


Figure 9. k_{-L} is independent of solids loading.

They identified two bubble classes, rigid (small) or elastic (large) bubbles. They observed that k_L is constant within each bubble class, is smaller for rigid bubbles and varies linearly with bubble size in the transition region between classes. If the addition of solids were causing bubble coalescence and concomitant bubble diameter increases, but only within one class of bubbles, both observations in this study would be explained. The value of a would decrease while that of k_L would remain constant.

No successful theoretical framework has been proposed to encompass mass transfer in three phase slurry reactors. Joosten et al. (1977) attempted to correlate variations in $k_L a$ due to increased solids loading in a mechanically agitated vessel with apparent viscosity, the bulk property of the slurry which changes the most. However, while this correlation allowed them to explain differences in particle size upon $k_L a$, it allowed no simplification for particle density or composition differences.

The results of this study indicate that such a simplification may be deceptive. Theories for mass transfer propose that k_L is proportional to diffusivity raised to a power between 1/2 and 1. Diffusivity is in turn inversely proportional to viscosity. Converted to a relative viscosity basis, one would thus anticipate that k_L would decrease with increased solids loading. The fact that k_L in this study instead was constant serves to clarify the difference between macroscopically and microscopically measured fluid properties. Over the length scale important for diffusion (and hence mass transfer), no effect of particle concentration upon mass transfer would be anticipated.

Additionally, experiments measuring the change in mass transfer parameters due to increases in viscosity yield differing results depending

upon whether the viscosity increase is caused by the addition of solids or by the addition of a high viscosity fluid. Sharma and Mashelkar (1968) and Abdel-aal et al. (1966) demonstrated an increase in α with increased viscosity for a glycerol and water mixture in a bubble column. Godbole et al. (1983) and Collakota and Quin (1984), however, reported that α decreased with increased viscosity caused by increased solids loading. Clearly, the mechanism for bubble coalescence and breakup depends upon how the viscosity increase is brought about.

Godbole et al. (1983) have attributed the changes observed in α with increased solids loading for different solid compositions to wetting properties of the solids. This was done for an aqueous sulfite solution in a bubble column. However, in the present study, all particles used were completely wet by the organic solution. This suggests that another mechanism must be operating to cause the reduced interfacial area with inert solids loading.

Interfacial area is determined by the extent of both bubble breakup and subsequent bubble coalescence. The orifice Reynold's number ($N_{Re,O} = 4Q_{D,g}/\pi d_o \mu_g$) is the key parameter characterizing the amount of kinetic energy in the gas available for bubble breakup at the exit from the orifice (Bhavaraju et al., 1978). For the high $N_{Re,O}$ used in this study, Bhavaraju et al. (1978) have shown that bubble breakup is independent of the properties of the liquid. Consequently, a marked effect upon bubble breakup is not expected by increased solids loading. An increase in bubble coalescence due to increased solids loading is thus hypothesized as the cause of reduced interfacial area. Perhaps a cage effect is occurring. As bubbles move toward each other the concentration of solids immediately surrounding the bubble pair could increase. The resistance this cage would

provide could increase bubble contact time and enhance coalescence probability. The cage of particles around colliding bubbles would provide the major mechanism for bubble coalescence to which decreased interfacial area is attributed. Wetting differences would provide a secondary mechanism. The presence of solid, nonwetting particles could induce bubble coalescence via a bridging mechanism. As two bubbles approach, particles in the film between them or attached partially to one bubble or another could at some point contact both bubbles. The lowered surface tension at the bubble and particle interfaces would provide a mechanism for increasing bubble contact time and would thus enhance coalescence probability. This additional mechanism would explain why polypropylene particles exhibited a greater rate of area decrease at higher solids loading.

Conclusions

The overall mass transfer coefficient and the interfacial area decrease linearly with inert solids loading in a mechanically agitated vessel. The decrease in $k_L a$ can be attributed to the decrease in a : k_L is independent of solids loading for the conditions of this study.

Both $k_L a$ and a are independent of particle size in a mechanically agitated vessel for particle sizes below 300 μm . Both are also independent of particle density. Stirring speed is shown to be an appropriate basis for comparison of mass transfer parameters.

At high solids loadings $k_L a$ and a become dependent upon particle composition. Particles which are more nonwetting cause a larger decrease in $k_L a$ and a .

NOMENCLATURE

a	measured interfacial area per volume of slurry (m^2/m^3 solid + liq.)
a_{CSTR}	calculated interfacial area assuming CSTR in the gas phase (m^{-1})
a_{PER}	calculated interfacial area assuming PER in the gas phase (m^{-1})
A	gas phase species in reaction (usually CO_2)
$[A^*]$	saturation concentration of A in liquid ($\text{kg.mol}/m^3$)
B	liquid phase species in reaction (usually CHA)
$[B_0]$	bulk concentration of B ($\text{kg.mol}/m^3$)
CHA	cyclohexylamine
$[CHA_0]$	bulk concentration of CHA ($\text{kg.mol}/m^3$)
$[CHA_0]_0$	bulk concentration of CHA at $t=0$ ($\text{kg.mol}/m^3$)
$[\text{CO}_2^*]$	saturation concentration of CO_2 in liquid ($\text{kg.mol}/m^3$)
$[\text{CO}_2^*]_{\text{eq}}$	concentration of CO_2 in equilibrium with amine ($\text{kg.mol}/m^3$)
C	concentration of solute in bulk ($\text{kg.mol}/m^3$)
$C_{g,o}$	concentration of helium gas exiting the reactor ($\text{kg.mol}/m^3$)
C_g^*	concentration of helium gas inside the reactor ($\text{kg.mol}/m^3$)
$C_{g,\text{ave}}$	average concentration of helium gas inside the reactor ($\text{kg.mol}/m^3$)
C_i	concentration of solute at the interface ($\text{kg.mol}/m^3$)
C_L	concentration of helium in liquid ($\text{kg.mol}/m^3$)
$C_{L,\text{sat}}$	helium saturation concentration in the liquid ($\text{kg.mol}/m^3$)
d_f, d_i, d_o, d_t	diameter of baffle, impeller, orifice, tank (m)
$D_A, D_B, D_{\text{CHA}}, D_{\text{CO}_2}$	diffusivity of A, B, CHA, CO_2 , helium, nitrogen in the liquid (m^2/s)
G	molar flow rate of inerts (usually N_2) ($\text{kg.mol}/s$)
H	Ostwald coefficient for helium (ml dissolved gas/ml liquid)
H^*, H_H^*	Henry's law constant for CO_2 or O_2, H_2 ($\text{kg.mol solute}/\text{MPa}\cdot m^3 \text{ liq.}$)
k_{FT}	first order kinetic rate constant for Fischer-Tropsch rxn. (s^{-1})
k_g	gas phase mass transfer coefficient ($\text{kg.mol}/m^2\cdot s\cdot \text{MPa}$)
k_L	liquid phase mass transfer coefficient (m/s)
$k_{L,H}$	liquid phase k_L for hydrogen (m/s)
$k_{L,a}$	overall mass transfer coefficient (s^{-1})
k_m	rate constant for irreversible reaction: mth order in $[A^*]$, nth order in $[B_0]$ ($m^{3m+3n-3}/\text{kg.mol}^{m+n-1}\cdot s$)
k_{12}	rate constant for irreversible reaction: 1st order in $[\text{CO}_2^*]$, 2nd order in $[\text{CHA}_0]$ ($m^6/\text{kg.mol}^2\cdot s$)

m	order of reaction in gas phase component
M	Hatta number = $2D_A k_{Am} [A^*]^{m-1} [B_0]^n / [(m+1)k_L^2]$
n	order of reaction in liquid phase component
NDIR	nondispersive infrared analyzer
N_m	mass flux ($\text{kg.mol/m}^2\text{s}$)
$N_{Re,O}$	orifice Reynold's number = $4Q\rho_g / \pi d_o \mu_g$
P	partial pressure of solute in bulk (MPa)
P_i	partial pressure of solute at the interface (MPa)
P_H	partial pressure of hydrogen in the reactor (MPa)
P	total pressure in the reactor (MPa)
Q	purge or reaction gas flow rate (m^3/s)
R	recycle ratio = flow of recycle/flow of fresh feed (-)
R_k	kinetic reaction rate of CO_2 and CHA ($\text{kg.mol/m}^3\text{s}$)
R_r	rate of transfer of A or CO_2 into the liquid ($\text{kg.mol/m}^2\text{s}$)
SIP	standard temperature and pressure (0.101 MPa, 20 °C) for gas flow rates and physicochemical properties, unless specified
S_v	space velocity of inlet H_2 and CO ($\text{kg.mol/m}^3\text{s}$)
St_m	Stanton number (Table IV.2) = $aV_G^{-1} [D_A k_{Am} [B_0]^n (H^*P)^{m+1}]^{1/2}$
t	time (s)
TC	thermal conductivity
T	temperature (K)
u_g	superficial gas velocity = Qh/V (m/s)
V	volume of ungasged slurry (m^3)
x_i	moles of A inlet feed/moles inert inlet feed (-)
x_i'	moles of A inlet plus recycle/moles inert inlet plus recycle (-)
x_o	moles of A outlet/moles of inert outlet (-)
X	conversion (-)
y_i	mole fraction of A in inlet feed (-)
y_o	mole fraction of A in outlet (-)
z	stoichiometric coefficient relating $[A^*]$ to $[B_0]$ (-)
ϵ_g	gas holdup = volume gas/volume ungasged slurry (-)
ϵ_s	solids holdup = volume solids/volume ungasged slurry (-)
μ_g	gas viscosity ($\text{kg/m}\cdot\text{s}$)
ρ_g	gas density (kg/m^3)

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