

GAS-LIQUID-SOLID OPERATION OF A VORTEX-INGESTING STIRRED TANK REACTOR

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The vortex-ingesting stirred tank reactor uses a combination of a helical screw impeller and concentric draft-tube to entrain and recycle headspace gas and disperse it as bubbles in a liquid reaction solvent. This study presents an experimental characterisation of a 0.29 m diameter vortex-ingesting gas-liquid stirred reactor, operated with solids mass fractions from 0 to 25%, over a range of specific power inputs up to 3 W/kg and for a variety of tank geometries. As was found in previous studies the most important geometric variable is the draft-tube top clearance, since this has the most direct effect on the gas ingestion rate. The presence of low solids fractions causes a significant increase in the minimum speed required for vortex ingestion of the gas. At low impeller speeds and low solids mass fractions, accumulation of particles in the annulus surrounding the draft-tube reduces the pumping capacity of the impeller. This mechanism also reduces the gas holdup and gas-liquid mass transfer coefficient, although not always in a monotonic fashion. In this design of reactor, aeration results in only a small decrease in the specific power input and so the just-suspended condition is not much affected by the presence of gas.

Keywords: Stirred tank reactor, gas-liquid-solid mixing, mass transfer

INTRODUCTION

Industrially, stirred tank reactors are widely used to perform three-phase operations involving dispersion and dissolution of a reactant gas into a continuous liquid phase; typically, the solid may be present as a heterogeneous catalyst, or as a particulate product of a chemical reaction. For example, terephthalic acid production involves liquid phase air-oxidation of *p*-xylene in a solvent in the presence of a cobalt catalyst; the product is also a solid, which is removed as a slurry. Some other examples of commercially important reaction processes relying on gas-liquid-solid dispersion in stirred reactors include fermentation, hydrogenations, chlorination and alkylations. In conventional stirred reactors, the gas is continuously sparged into the vessel and is dispersed by one or more rotating impellers, which also serve to generate liquid flows for blending and particle suspension. Often only a small fraction of the feed gas is absorbed in a single pass and the remainder disengages into the headspace of the reactor. In some cases (*e.g.* with air sparging), these headspace off-gases may be safely vented or scrubbed, but in other cases, the reactant gas is valuable, or hazardous, and must be recycled back through the sparger. Ideally, such a reactor should be designed to operate in a "dead-end" mode with respect to the reactant gas, *i.e.* the majority of the disengaging gas should be recycled until it is all absorbed in the reaction solvent.

Recycling the headspace gases via an external recompression loop is one solution, but it involves additional capital and running costs. When the gas is hazardous to compress (*e.g.* oxidations with pure O₂, or hydrogenations), external loops should be avoided and there are advantages in using a type of self-inducing gas-liquid reactor, which features internal recycling of the headspace gas¹. Self-inducing systems include: (i) surface aeration in a partially baffled, or unbaffled, vessel²; (ii) gas-induction through a hollow-bladed shaft and impeller³; (iii) self-induction through a rotor-stator device and concentric standpipe⁴; or, (iv) ingestion of gas through the formation of surface vortices^{1,5}. The reviews by

Patwardhan^{4,6} have categorised and described the various types of self-inducing impeller systems.

Thus, self-inducing devices are a safer alternative for reactions involving hazardous gases hazardous requiring compression; as closed systems, they minimise the risk of leaks, when dealing with toxic or expensive solute gases. In three-phase mixing operations, such as hydrometallurgical processes, the blockage of gas spargers by solid particles is a common issue, which can be avoided using a self-inducing device. The main drawbacks of self-inducing devices compared to sparged systems are (i) the loss of operating flexibility, as the gas flow rate depends on the impeller speed and can no longer be varied independently; (ii) the induced gas flow rate may be sensitive to changes in the liquid level; (iii) large specific power inputs may be required to effect induction of the gas, at large scale.

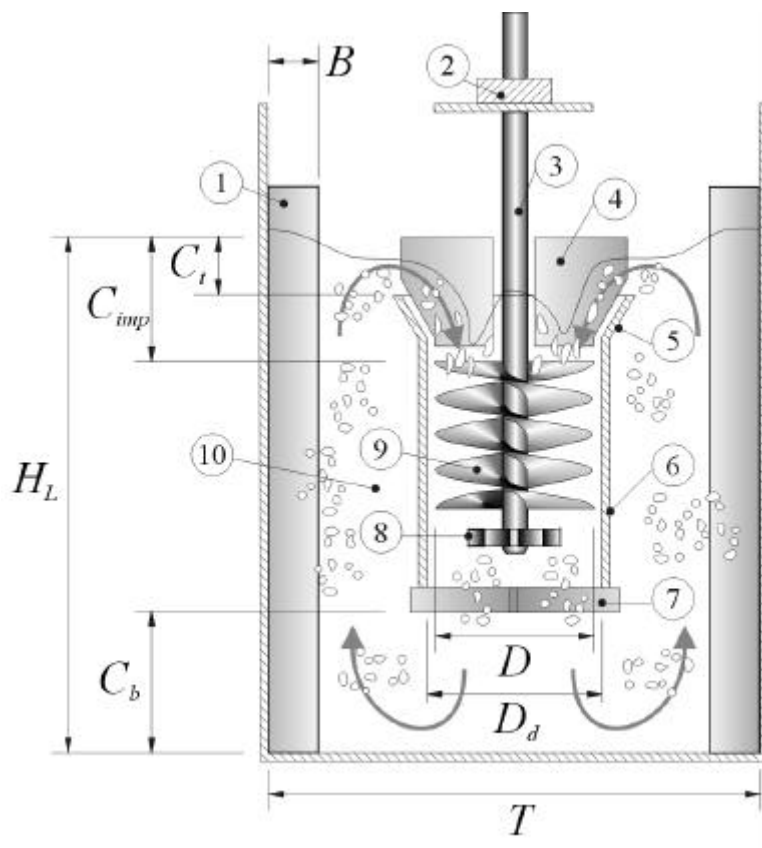


Fig. 1 Schematic diagram of the AGR experimental apparatus. 1: wall baffles, 2: motor and shaft bearing, 3: impeller shaft, 4: upper cruciform baffles, 5: draft-tube flare, 6: draft-tube, 7: lower cruciform baffles, 8: radial bladed turbine, 9: helical screw impeller, 10: annulus

THE VORTEX-INGESTING STIRRED TANK REACTOR

One example of a vortex ingesting system is the Advanced Gas Reactor (AGR), developed and patented by Praxair⁷⁻¹⁰. Fig. 1 shows a typical AGR configuration, employing a combination of two impellers and a concentric draft-tube to entrain and disperse gas from the headspace. The main impeller is a four-flight down-pumping helical screw impeller; a secondary impeller, a six flat bladed turbine, is attached to the same shaft and generates additional shear for bubble breakage. The draft-tube is cylindrical, with a conically-flared top section, which provides a streamlined flow entry into the draft-tube.

The helical screw generates a strongly swirling liquid down flow, which is redirected axially by the lower cruciform baffles. The discharge flow then impinges on the base of the vessel and is deflected upwards into the annulus surrounding the draft-tube. At the top of the annulus, the up flow re-enters the draft-tube, flowing around the top cruciform baffles. Above a certain minimum impeller speed, these baffles promote the formation of surface vortices through which headspace gas is ingested. Gas is drawn into the liquid through the surface vortices and enters the suction side of the impeller, where it is dispersed as fine bubbles within the draft-tube. These bubbles are then circulated along with the liquid flow through the outer annulus. The strong liquid flow over the flared entry of the draft-tube causes a significant fraction of the bubbles in the annulus to be recirculated, leading to longer gas residence times. The remaining fraction of bubbles, in the annulus flow, disengages at the free surface, but subsequently in a closed vessel this gas is recycled by vortex ingestion, so that the reactor can be operated in a “dead-end” mode with respect to the feed gas.

Previous studies^{1,11} have investigated the AGR as a gas-liquid contacting device and have shown that the mass transfer coefficients obtained are as good as with conventional sparged systems, at the same specific power input and gas superficial velocity. Many speciality chemical reactions also involve suspension of a heterogeneous catalyst and so the current study has investigated gas-liquid-solid operation of the AGR. In this paper, results are reported for the effects of solids mass fraction on (i) the minimum speed for vortex ingestion, (ii) the gas holdup and (iii) the volumetric mass transfer coefficient; additionally, the effects of gas ingestion on the just-suspended impeller speed were also studied.

EXPERIMENTAL

The apparatus shown in Fig. 1 comprised a flat-bottomed cylindrical tank, of 0.29 m internal diameter, fitted with four evenly spaced wall baffles, of width equal to one tenth the vessel diameter. The concentric draft-tube, had a cylindrical main body, with a diameter equal to the third of the vessel diameter and a conically flared top section, forming the liquid inlet. At its entry and exit, the draft tube was also fitted with cruciform baffles. The mixing vessel and all its internals, including the draft tube, were made out of Perspex for optical access. Two separate impellers were mounted on the shaft: (i) a downward pumping, four flight helical screw, and (ii) a six flat bladed turbine. The main impeller diameter was $D/D_d = 0.90$, giving a small draft-tube/impeller clearance to ensure effective downward pumping. Complete dimensions of the baffles and impellers are given in Table 1 below.

The immersion depths of both impellers could easily be varied by sliding them up or down along the shaft, whilst maintaining all other heights or clearances. This practical convenience allowed the investigation of the separate effects of the geometric ratios C_t/T , C_{imp}/T and H_L/T on the gas-liquid-solid flow characteristics. To minimise shaft vibrations, the bottom end of the shaft was inserted into a central PTFE bottom bearing (not shown).

Agitation was provided through a 0.75 kW motor, with a calibrated speed controller and digital display; the specific power input could be varied between about 0.01 and 3.2 W/kg and the Reynolds numbers were in the range 2.6×10^4 and 1.7×10^5 , so that the working flow regime was always fully turbulent. The vessel headspace was open to atmosphere and the test fluids were air and tap water. Air was supplied by surface aeration only.

Vessel	Impellers	Draft tube
Diameter $T=0.29$ m Baffle width: $B = 0.029$ m Liquid heights: $1.1 < H_l / T < 1.4$ Top clearances: $0.06 < C_t / T < 0.16$ Bottom clearances: $0.26 < C_b / T < 0.49$ Impeller submergence: $0.24 < C_{imp} / T < 0.44$	<i>Upper:</i> 4 flight helical screw Diameter $D = 0.0875$ m Height: 0.066 m Pitch: 1.5 <i>Lower:</i> 6 flat blades Diameter: 0.071 m Blade height: 0.01 m Blade width: 0.019 m Blade thickness: 0.0031 m	<i>Main body:</i> Diameter: $D_d = 0.097$ m Height: 0.2 m <i>4 Top baffles:</i> Height: 0.075 m Top width: 0.038 m <i>4 Bottom baffles:</i> Height: 0.05 Width: 0.031 m <i>Flare:</i> Top diameter: 0.115 m Height: 0.032 m Angle: 15° to vertical

Table 1 Geometry and dimensions of the experimental rig

The minimum speed for aeration, N_a , corresponds to the onset of vortex ingestion, and is defined to be the condition at which gas is first entrained through the breakage of shallow gas vortices at the free liquid surface and enters the impeller down flow. The minimum speed for aeration was determined visually and every measurement was repeated many times so that an average value of N_a and the associated experimental error could be estimated. Close to the minimum speed for aeration, a few bubbles are sporadically ingested, but in this work it was decided to define N_a to correspond to the condition when regular and stable ingestion of gas was observed. To avoid the effects of hysteresis, the value of N_a was always determined whilst increasing the rotational speed from zero.

The impeller torque was measured using shaft-mounted strain gauges and a four-way slip ring assembly to a filter and amplifier, coupled to an averaging voltmeter. The system was calibrated by applying pre-set loads to the shaft and at each impeller speed, the corresponding no-load torque was subtracted from the measured values to allow for friction in the bottom bearing. At impeller speeds below 200 rpm, the errors from subtracting the no load power were unacceptably high and so measurements were only conducted at higher speeds.

Average gas holdup values were obtained by measuring the increase in aerated level at speeds in excess of N_a . The free surface above the flared draft tube is highly deformed by the gas-ingesting vortices and the level in this region is difficult to determine accurately. However, in the annulus region, which includes almost 90% of the tank volume, the liquid level is almost flat. Thus, average gas holdups are based on measurements of the annulus liquid level, H_a , and the unaerated liquid height, H_l , using eq.(1), with negligible error resulting from the depressions caused by vortex ingestion.

$$e_g = \frac{(H_a - H_l)}{H_a} \quad (1)$$

Three-phase experiments were performed using glass ballotini particles with a Sauter mean diameter of 990 μm and a standard deviation of about 100 μm , at solids loadings from 0 to 25% by mass. The solid density was found to be 2960 kg/m^3 , using a helium pycnometer. Initial observations revealed that the down flow jet issuing from the draft-tube is effective at suspending particles. For given a impeller submergence and draft-tube clearance and with a

known mass fraction of particles loaded, the impeller speed was increased from zero until the just suspended condition was reached, as determined by Zwietering's '1-2 second' criterion¹². This procedure was repeated, to obtain an average value of N_{js} and an estimate of the uncertainty. The gas holdup, N_a and N_{js} experiments were conducted over a range of draft-tube top clearances, liquid heights and impeller submergences, as detailed in Table 1.

Volumetric mass transfer coefficients, k_{la} , were determined from unsteady-state experiments using the absorption of oxygen from air bubbles ingested by the surface vortices. Initially the, water was deoxygenated by sparging nitrogen supplied through an open-ended pipe immersed in the draft-tube and with the impeller operating at a speed below N_a . The dissolved oxygen percentage saturation was measured using a polarographic oxygen electrode. When the concentration approached zero, the impeller was preset to rotate at the required speed and then switched off; the nitrogen supply was removed. After allowing nitrogen to disengage, the impeller was restarted and as air was almost immediately entrained, the dissolved oxygen concentration began to rise. Initially, ten experiments were repeated at $N = 2N_a$, with no particles present, in order to make an estimate of the repeatability and error. The geometry used was $H_l/T = 1.25$, $C_{imp}/T = 0.4$, $C_t/T = 0.09$ and $C_b/T = 0.31$ and experiments were conducted over a range of impeller speeds, when no particles were present. Solid particles were then loaded into the tank and the procedure was repeated over the range of solid mass fractions.

The results were analysed by assuming that both the gas and liquid phases could be treated as well mixed¹. Oxygen is sparingly soluble (hence $k_l \approx K_l$) and only small amounts are absorbed, so that the oxygen partial pressure in the bubbles remains almost constant. Writing a mass balance on dissolved oxygen in the liquid phase yields

$$V \frac{dC(t)}{dt} = V k_{La} (C^* - C(t)) \quad (2)$$

where $C(t)$ is the dissolved oxygen (DO) concentration at time t , V is the liquid volume (approximately equal to the aerated volume at low gas holdups) and C^* is the dissolved oxygen concentration in equilibrium with the gas. Step change tests revealed that the DO probe had an approximately first-order response with a time constant, τ , of about 4 s. Thus the measured probe concentration, $C_p(t)$ may be related to $C(t)$ by

$$\frac{dC_p(t)}{dt} = (C(t) - C_p(t)) / \tau \quad (3)$$

Simultaneous solution of eqs.(2) and (3), subject to the boundary conditions that $C(0) = C_p(0)$, yields

$$C_p(t) = C^* \left[1 - \frac{e^{-k_{La}t} - k_{La}\tau e^{-t/\tau}}{1 - k_{La}\tau} \right] \quad (4)$$

A *non-linear* least squares fit to eq.(3), for the known value of τ was performed to fit the volumetric mass transfer coefficient to the unsteady-state oxygen absorption data.

RESULTS AND DISCUSSION

MINIMUM SPEED FOR SURFACE AERATION

Results were obtained for the effects of solids mass fraction on the minimum speed for surface aeration, N_a , which are plotted in fig. 2 in terms of the aeration Froude number, defined by

$$Fr_a = \frac{N_a^2 D}{g} \quad (5)$$

With no solids present, the measured values of N_a were within 15% of those predicted by the correlation, $Fr_a = 22.5(C_t/T)^{1.58}$, proposed by Leguay¹. At higher solids loadings the minimum speed for aeration increased markedly to a maximum at about 15%, but thereafter N_a fell with increasing solids mass fraction. Observations showed that prior to aeration and at low solids mass fractions, the emulsion height in the annulus was between $0.6H_l$ and $0.7H_l$, so that no solids were circulated to the top of the annulus and none entered the draft-tube. Thus, there was a bulk density difference, and therefore a hydrostatic pressure difference, between the annulus and the draft-tube, which had to be overcome by impeller pumping. Any reduction in discharge coefficient Q/ND^3 , caused by the increased hydrostatic pressure difference due to the presence of solids in the annulus, would require a higher impeller speed N_a to first ingest gas via the surface vortices. At the higher solids mass fractions, a proportion of the particles were circulated into the draft-tube, generating a more uniform bulk density in the draft-tube and annulus sections and hence reducing the hydrostatic pressure difference to be overcome by the impeller. Thus, at the highest solids mass fractions, the minimum impeller speed for aeration returned to approximately the value for no solids being present.

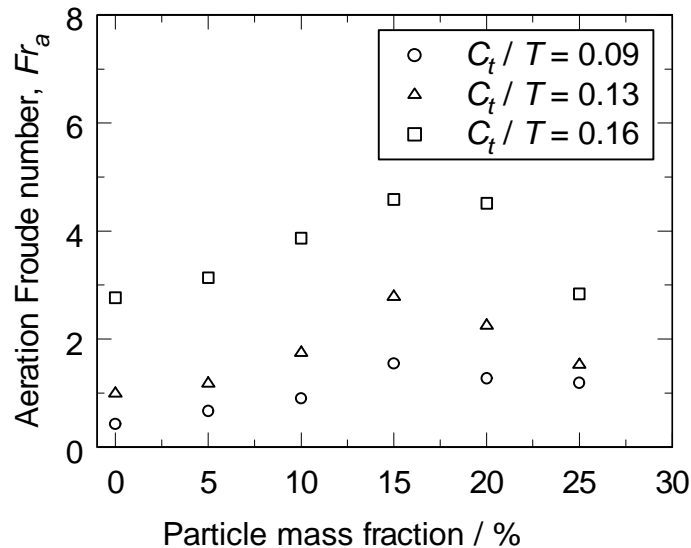


Figure 2 Minimum conditions for gas ingestion for various solids loadings and draft-tube top clearances at $H_l/T = 1.25$ and $C_{imp}/T = 0.4$

Figure 2 also illustrates the effect of the draft-tube top clearance on the minimum speed for aeration. At constant liquid height and impeller position, the value of N_a falls as the

top of the draft-tube is brought closer to the free surface, indicating that the strength and depth of the surface vortices is rather sensitive to C_t/T .

GAS HOLDUP

Figure 3 shows that the gas holdup is affected by the solids mass fraction, with the largest values of e_g being obtained when no particles are present. Similar results have been reported for three-phase operations in conventional sparged reactors: generally, as the solid concentration increases, the gas hold-up fraction decreases¹³⁻¹⁵. Chapman *et al.*¹⁶ concluded that in sparged tanks, provided the gas is completely dispersed and the solids are suspended, there is only a small effect of particles on the gas-liquid hydrodynamics. However, here the effect is more subtle and is non-monotonic, since the entrained gas flow rate depends on the ability of the circulating liquid flow to ingest gas from the headspace. The results of fig. 2 suggest that at low solids mass fractions the gas ingestion rate at a given impeller speed is reduced in the presence of solids and it is proposed that this process recovers at the highest solids loadings and with increasing N , as the solids become more uniformly distributed between the annulus and draft-tube regions. Thus in fig. 3, relative to the 0 % data, the initial effect of adding solids is to reduce the gas holdup; with increasing power input the difference between the 0% and 25% solids holdups has decreased. At low power inputs, the minimum holdup is obtained at a solids mass fraction of 15%, which corresponds to the maximum value of N_a in fig. 2; at higher solids %, the gas holdup increases, confirming the trend suggested by fig. 2.

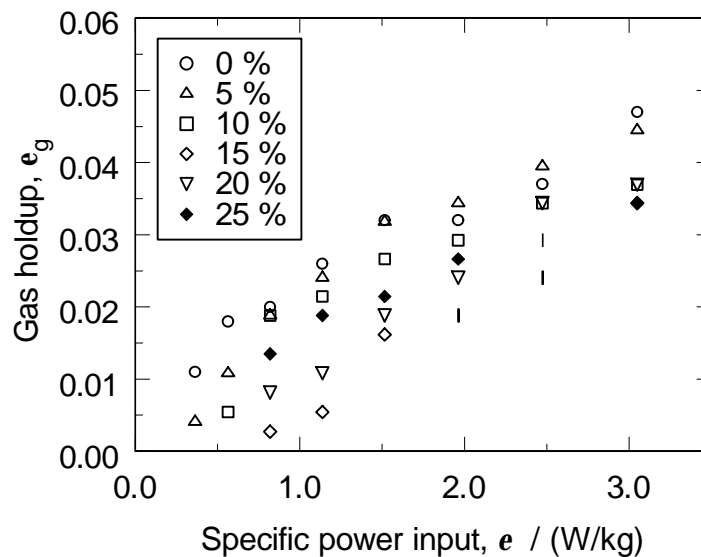


Figure 3 The effects of solids mass fraction on the mean gas holdup for $H_l/T = 1.25$, $C_b/T = 0.31$, $C_t/T = 0.09$ and $C_{imp}/T = 0.4$

JUST-SUSPENDED IMPELLER SPEED

Initially, experiments were conducted at high liquid levels, where there was no gas entrainment, to obtain the effects of varying the draft-tube bottom clearance on the just-suspended impeller speed, N_{js} (see fig. 4). When the draft-tube is brought too close to the base of the vessel (small values of C_b/T), the discharge flow from the impeller is choked and the particles require a high speed to be just-suspended. Visual observations of the just-

suspended condition were reproducible, since the last particles to become suspended always left from the upstream corner behind the wall baffles. Displacing the baffles a small distance from the wall and slightly off the tank base would result in lower values of N_{js} .

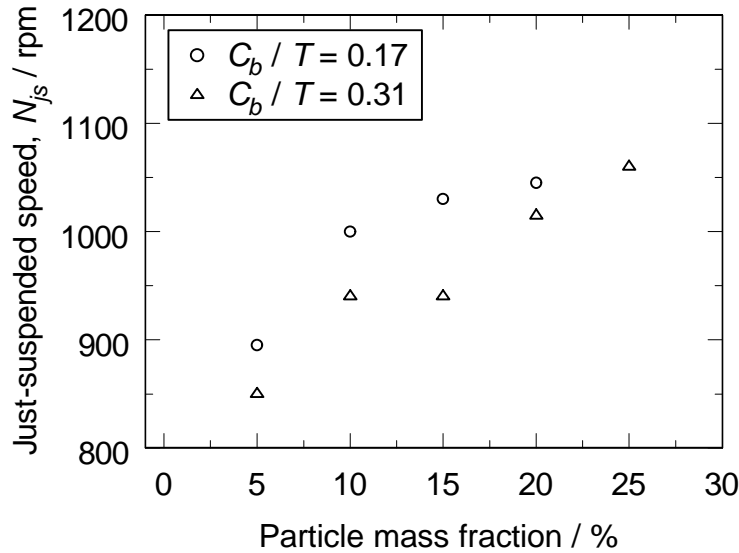


Figure 4 The effects of bottom clearance on the just-suspended impeller speeds under conditions of no aeration

$$H_l/T = 1.37, C_b/T = 0.17 \text{ and } 0.31, C_t/T = 0.23 \text{ and } 0.09 \text{ and } C_{imp}/T = 0.4$$

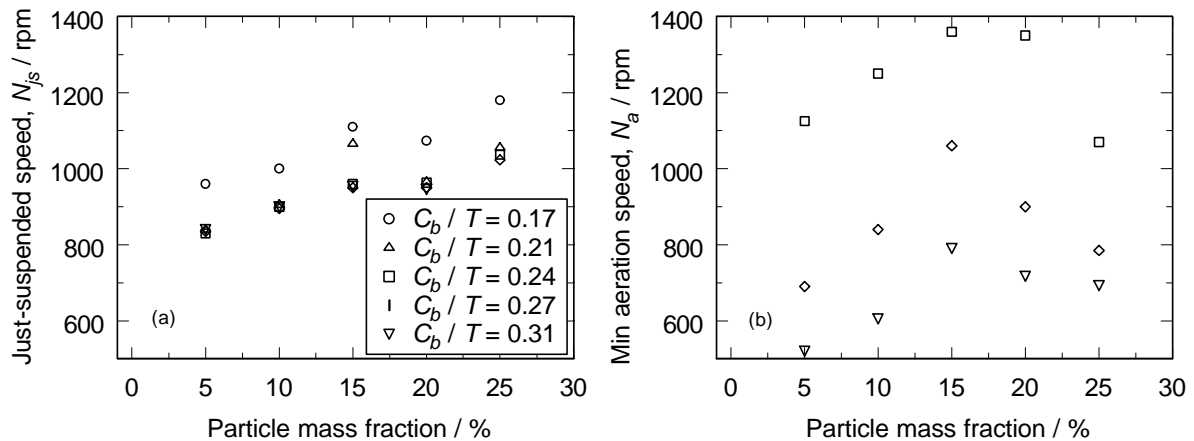


Figure 5 The effects of bottom clearance on (a) the just-suspended impeller speeds under aerated conditions and (b) the minimum speed for aeration for

$$H_l/T = 1.25 \text{ and } C_{imp}/T = 0.4. \text{ The same legend applies to both (a) and (b).}$$

Increasing the draft-tube clearance above $C_b/T = 0.21$, gave almost the same just-suspended speeds, as is shown in fig. 5(a), for a lower liquid height where aeration occurs more easily. For a constant H_l/T , the draft-tube top and bottom clearances are related via $C_b/T = H_l/T - 0.85$: at the lowest C_b/T , the draft-tube top clearance is large and the value of N_a is greater than N_{js} (compare figs 5(a) and (b)), so that suspension occurs into a liquid only flow. At the largest C_b/T , the draft-tube top clearance is small and the value of N_a is

less than N_{js} , so that suspension occurs into a gas-liquid flow. It is evident from fig. 5(a) that (i) N_{js} is relatively insensitive to C_b for clearances greater than $0.21T$ and (ii) the presence of gas has little effect on solids suspension. The latter may be attributed to the low gas holdups obtained in this system and to the relative insensitivity of the power input to aeration¹.

The effects of solids fraction on N_{js} may be compared with Zwietering's correlation¹²

$$N_{js} = S \dot{\epsilon}^{0.1} d_p^{0.2} \left(\frac{g \Delta \tilde{n}}{\tilde{n}_l} \right)^{0.45} X^{0.13} / D^{0.85} \quad (6)$$

Regression of the data in fig. 5(a) showed that the exponent on solids mass fraction, X , varied between 0.10 and 0.14 for the various values of C_b / T studied, in good agreement with the prediction of eq.(6). Values of the Zwietering parameter S of between 5.9 and 7.2 were obtained for the various bottom clearances of the AGR.

GAS-LIQUID MASS TRANSFER COEFFICIENT

The highest gas holdups were obtained at the lowest draft-tube top clearance and so this geometry was chosen to study the effects of solids on the mass transfer coefficient. The results shown in fig. 6 (a) and (b) show that, as expected, the gas-liquid mass transfer coefficient increased with increasing specific power input (or impeller speed) as a result of (i) increased levels of turbulence and (ii) increased volumetric flow rates of ingested gas. The mass transfer coefficients approximately follow a power-law relationship with specific power input of $k_{l,a} \propto \epsilon^{0.52}$, which compares reasonable well with Leguay's measurements¹ in a similar system which gave $k_{l,a} \propto \epsilon^{0.50}$.

Similar trends to fig. 6 have been observed by many previous workers^{14,17}. Joosten *et al.*¹⁸ reported that the decrease in $k_{l,a}$ with solid loading was due to the increased apparent viscosity of the liquid-solid mixture, which enhanced bubble coalescence, thus decreasing the interfacial area. In contrast however, Chapman *et al.*¹⁹ noted only small differences in $k_{l,a}$ at low particle concentrations, but a substantial decrease at 20% solids mass fraction; Mehta and Sharma²⁰ found the mass transfer coefficient to decrease initially, which they explained to be due to the particles decreasing surface mobility.

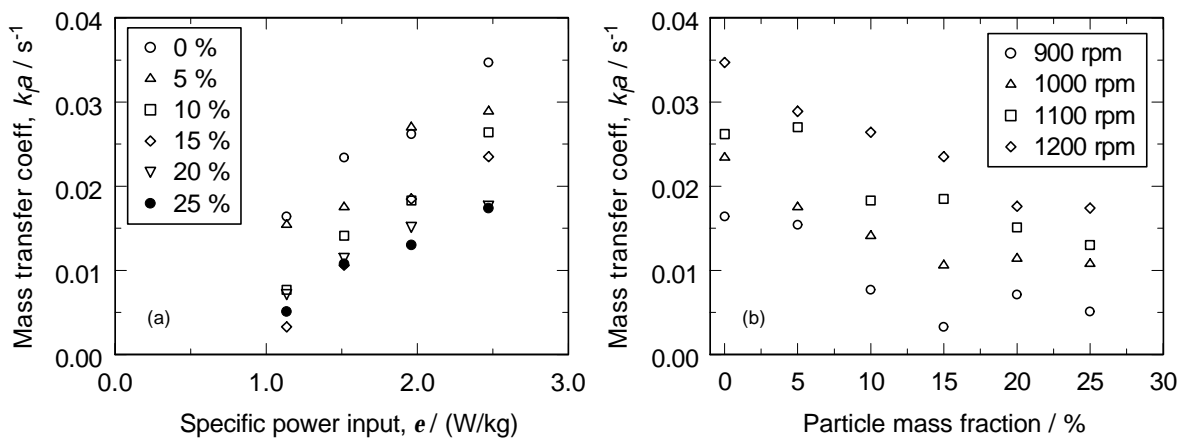


Figure 6 The effects of solids loading the volumetric mass transfer coefficient on (a) as a function of specific power input and (b) at constant impeller speed

$$H_l / T = 1.25, C_b / T = 0.31, C_t / T = 0.09 \text{ and } C_{imp} / T = 0.4$$

In fig. 6, the presence of solids reduces the mass transfer coefficient, although the differences between 15% and 25% solids mass fraction are not large. As was explained in the discussion of the gas holdup and N_a measurements, the presence of solids which accumulate in the annulus, reduces the liquid circulation flow rate and hence the flow rate of ingested gas also is reduced, resulting in a lower interfacial area. This would seem to be the primary mechanism by which the volumetric gas-liquid mass transfer coefficient is reduced initially by the presence of solids in a vortex ingesting gas-liquid-solid reactor. A secondary effect is that turbulence damping¹⁹ by the particles results in a lower value of k_l .

CONCLUSIONS

This preliminary study is the first to report on the operation of the AGR as a three-phase (gas-liquid-solid) reactor. Unlike many self-inducing agitators, the AGR is well suited to solids suspension, since the discharge flow from the impeller directly impinges on the base of the tank, picking up particles and transporting them into the annulus. Other designs, such as the gas-inducing impeller³ have trouble in simultaneously suspending solids and ingesting a gas flow at reasonable specific power inputs.

In the results reported here, there is an effect of solids on the minimum speed for aeration, gas holdup and mass transfer coefficient, whereas aeration was found to have a negligible effect on N_{js} . Generally, the presence of solids makes gas-liquid operation more difficult. At low solids mass fractions, the particles are partially suspended in the annulus, increasing the hydrostatic pressure difference between the annulus and draft-tube and decreasing the liquid circulation velocities. In turn this affects the strength and depth of the surface vortices, which determine the minimum speed for aeration. At $N > N_a$, it is proposed that the flow rates of ingested gas are also reduced by this mechanism. At higher solids fractions and with increasing impeller speed, the solids distribution between the annulus and draft-tube is more uniform and there is a reduced effect of solids on the gas holdup and the mass transfer coefficient. Detailed examination of the effects of solids on the liquid discharge flow rate and volumetric rate of gas ingestion were not obtained here, but could be measured using the techniques developed by Leguay¹. These future experiments should be conducted to confirm the findings of the current work.

NOMENCLATURE

a	specific interfacial area (m^{-1})	H_l	liquid height (m)
B	baffle width (m)	k_l	liquid phase mass transfer coeff (m/s)
$C(t)$	DO concentration (kg/m^3)	N	impeller speed (rps)
$C_p(t)$	probe DO concentration (kg/m^3)	N_a	minimum speed for aeration (rps)
C_b	draft-tube bottom clearance (m)	N_{js}	just-suspended impeller speed (rps)
C_{imp}	impeller submersion depth (m)	V	liquid volume (m^3)
C_t	draft-tube bottom clearance (m)		
D	helical screw impeller diameter (m)	<i>Greek</i>	
D_d	draft-tube diameter (m)	e	power input per unit mass (W/kg)
Fr_a	Froude number at min. aeration (-)	e_g	gas holdup (-)
g	gravitational acceleration (m/s^2)	t	probe time constant
H_a	aerated height in the annulus (m)		

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