

# MASS TRANSFER AND HYDRODYNAMIC CHARACTERISTICS OF A LONG DRAFT TUBE SELF-INGESTING REACTOR (LDTSR) FOR GAS-LIQUID-SOLID OPERATIONS

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## ABSTRACT

Gas-liquid stirred vessels are widely employed to carry out chemical reactions involving a gas reagent and a liquid phase. The usual way for introducing the gas stream into the liquid phase is through suitable distributors placed below the impeller. An interesting alternative is that of using “self ingesting” vessels where the headspace gas phase is injected and dispersed into the vessel through suitable surface vortices. In this work the performance of a Long Draft Tube Self-ingesting Reactor dealing with gas-liquid-solid systems, is investigated. Preliminary experimental results on the effectiveness of this contactor for particle suspension and gas-liquid mass transfer performance in presence of solid particles are presented. It is found that the presence of low particle fractions causes a significant increase in the minimum speed required for vortex ingestion of the gas. Impeller pumping capacity and gas-liquid mass transfer coefficient are found to be affected by the presence of solid particles, though to a lesser extent than with other self-ingesting devices.

## 1. 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 where, typically, solid particles may be present as a heterogeneous catalyst, as a reagent, or as a product of the chemical reaction. The usual way for introducing the gas stream into the liquid phase is through suitable distributors placed below the impeller [1]. An interesting alternative is that of using “self-inducing” impellers [2] or “self-ingesting” vessels [3-4] where, provided that the impeller is operated at sufficiently high rotational speeds, the gas phase from the headspace is injected and dispersed into the liquid either through an hollow shaft, or through suitable surface vortices. These systems have the advantage that they can be operated in “dead-end” mode so that practically all of the feed gas may be absorbed in the liquid, a useful feature whenever reactions involving a pure gas are to be carried out (e.g. hydrogenations, chlorinations etc.). Self-inducing devices are a convenient and safer alternative for reactions involving hazardous gases (toxic or flammable) as do not require any external circuitry for gas recycle and therefore they minimize the risk of leaks. Also, in three-phase mixing

operations the blockage of gas spargers by solid particles is a common issue, which can be avoided using a self-inducing device.

In this work the performance of a Long Draft Tube Self-ingesting Reactor (LDTSR) dealing with gas-liquid-solid systems, is investigated. The LDTSR main features are a high aspect-ratio and a fairly narrow multiple-impeller draft tube, through which the gas phase is ingested and drowned down to the vessel bottom, where it is finely dispersed into the slowly rising solid-liquid mixture. In some way, it may be regarded as a three-phase self-ingesting bubble column.

## 2. EXPERIMENTAL

The investigated reactor is depicted in Fig.1. It consisted of a flat bottomed cylindrical tank with an internal diameter of 280 mm and an height of 1450 mm, fitted with four evenly spaced baffles supporting a concentric draft tube with an internal diameter of 85 mm and length of 1200 mm (fitted in turn with four 10 mm wide internal baffles) and off-spaced from vessel bottom by 50 mm. Inside the draft tube five identical pitched blade impellers of diameter 0.065 m were mounted on the 17 mm dia. shaft, at 50, 150, 450, 750, 1050 mm from the upper brim of the draft tube. When the stirrer was operated at agitation speeds higher than a minimum impeller speed for gas ingestion, gas was continuously withdrawn from the headspace via the pronounced vortex, resulting in a two-phase mixture that was driven down through the draft-tube. Immediately below the draft-tube a 0.095 m diameter six-flat-blade hub-mounted turbine broke the gas phase into tiny bubbles that subsequently rose slowly in the annular portion of the vessel.

The minimum speed for aeration 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. In this work the minimum speed for aeration was considered to be attained only when regular and stable gas ingestion was observed.

Power consumption measurements were carried out by monitoring the temperature rise due to the agitation power input [4]. All temperature dynamics showed a remarkably constant slope. For simplicity, in the computation of power dissipation the heat capacity of vessel walls, shaft and impellers were neglected in front of that of the water mass. This allows to directly converting the observed temperature rise velocity (i.e.  $3.88 \cdot 10^{-4} \text{ }^\circ\text{C/s}$  at 900 rpm) into the relevant specific dissipation rate ( $1620 \text{ W/m}^3$  of liquid phase). It may be guessed that the underestimation of the power consumption incurred because of the quoted simplification accounts for only few percent, being therefore acceptable for most engineering purposes. The total agitation power was finally estimated by multiplying the specific power dissipation by the water volume in the system (0.0769 m<sup>3</sup>).

Average gas hold-up values were estimated from the clear liquid height above tank bottom at no agitation conditions and the levels under agitation conditions in the annular section and the draft tube determined by visual inspection of the liquid level. The following equation was used:

$$\varepsilon = \frac{\frac{\pi d_{ext}^2}{4} (h_{ext} - h_{rif}) - \frac{\pi d_{int}^2}{4} (h_{ext} - h_{int})}{\left( \frac{\pi d_{ext}^2}{4} (h_{ext} - h_{rif}) - \frac{\pi d_{int}^2}{4} (h_{ext} - h_{int}) \right) + \frac{\pi d_{ext}^2}{4} h_{rif}} \quad (1)$$

where  $h_{rif}$  is the clear liquid height above tank bottom at no agitation conditions,  $h_{ext}$  and  $h_{int}$  are the levels under agitation conditions in the annular section and the draft tube respectively while  $d_{ext}$  e  $d_{int}$  are the relevant diameters.

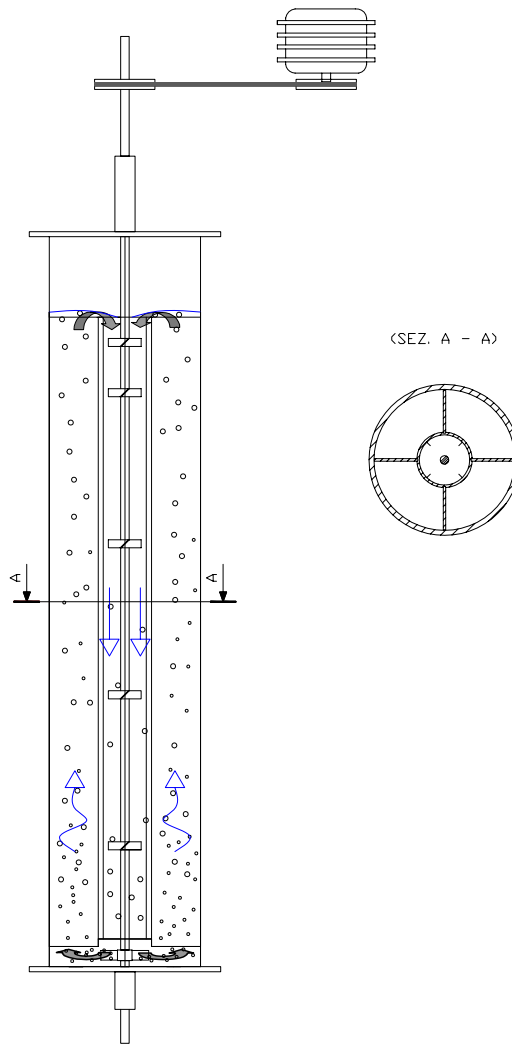


Figure 1. Schematic diagram of the Long Draft Tube Self-ingesting Reactor (LDTSR)

The volumetric mass transfer coefficient,  $k_L a$ , was assessed by unsteady state experiments by means of the simplified dynamic pressure method (SDPM) [5].

In this method the driving force for oxygen absorption is obtained by a sudden change of vessel pressure, with no need for sudden changes of gas phase composition above the liquid free surface. In particular, pressure inside the reactor was typically brought down to about 0.5 bar while stirring at medium agitation speeds to fasten the degassing process. Pressure and agitation were maintained for several minutes (typically 5 min) to allow for that the dissolved oxygen and nitrogen concentrations in the liquid phase to go down towards equilibrium with air at 0.5 bar (4.7 ppm wt for O<sub>2</sub> and 12.8 ppm wt for N<sub>2</sub>). Atmospheric pressure was then suddenly restored by admitting air to the reactor headspace while continuing stirring. Data acquisition was started shortly after the instantaneous pressure step change. A transitory followed, in which the difference between the equilibrium concentrations of both oxygen and nitrogen to air at 1 bar and the time dependent oxygen and nitrogen concentrations in the liquid phase decayed and eventually vanished. The dissolved-Oxygen concentration dynamics was measured via a suitable probe and the relevant time series was acquired by a PC. At the end of the transitory the stationary dissolved Oxygen concentration of about 9.3 ppm wt was reached inside the reactor. It is worth noting that with the simplified dynamic technique method (SDPM) the effect of the inert gas [4] is minimized and air absorption may be used [5].

Three-phase experiments were performed using glass ballotini particles (size range between 200 and 300  $\mu\text{m}$ , density = 2550  $\text{kg}\cdot\text{m}^{-3}$ ) at solids loadings from 0 to 1% by mass.

### **3. RESULTS AND DISCUSSION**

#### *3.1 Minimum speed for gas ingestion*

The minimum impeller speed for gas ingestion, as well as gas hold-up and mass transfer parameter were found to significantly depend on the liquid level above the brim of the draft tube. In particular larger minimum speeds and smaller gas hold-ups and mass transfer performances were observed the larger the level height above the brim. The choice of adopting a liquid height levelled with the draft-tube brim at no agitation conditions stemmed out from this consideration.

The presence of solid particles was found to strongly affect the minimum impeller speed for gas ingestion. In particular, stable hydrodynamic regimes, were observed at agitation speeds higher than about 700 rpm in absence of solid particles, while agitation speeds higher than 800 rpm were needed when even small amounts of solid particles were present in the system. In this last case, at 700 rpm, a large amount of gas phase is observable inside the draft tube but very few bubbles are able to reach the annular section where the gas hold-up is consequently very low. At 800 rpm the gas phase reaches vessel bottom where it is finally dispersed into the annulus in the rising solid-liquid mixture. An analogous increase in minimum gas-ingestion speed was observed by Conway et al. [3] in their AGR when it was used for three phase operations.

#### *3.2 Gas hold-up*

The hold-up values observed at the various agitation speeds are reported in Fig.2 for three different particle mass fractions. As it can be seen, gas hold-up increases while increasing agitation speed, in agreement with expectations, while the presence of suspended particles appears to have no influence on total gas hold-up.

However, visual observation of the reactor shows that the presence of particles modifies the gas hold-up distribution inside the reactor, sensibly reducing the gas fraction inside the annular section and increasing that in the down comer, though the total gas hold-up remains constant. This, in turn, may reduce the mass transfer efficiency of the system.

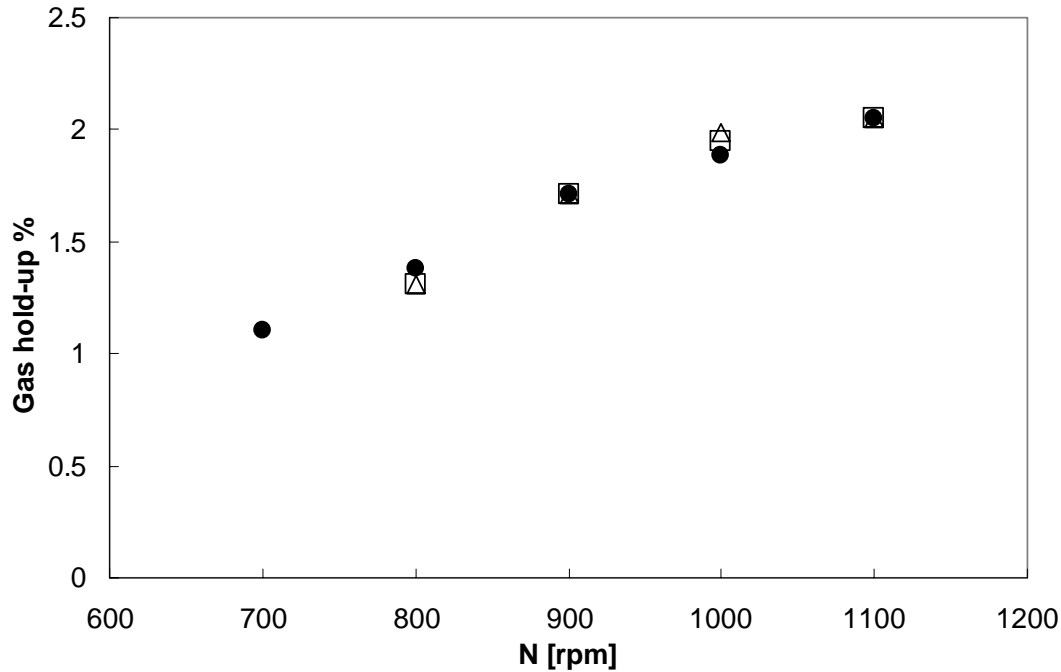


Figure 2. Total gas hold-up versus agitation speed for various particle mass fractions: solid symbols, particle mass fraction=0 wt%; open squares, 0.5 wt%; open triangles, 1 wt%

### 3.3 Power consumption

The specific power dissipation values obtained at the various agitation speeds by the temperature rise analysis [4] are reported in Fig.3. A steep increase of power dissipation with agitation speed is observed, as expected. As it can be seen, the addition of small amounts of solid particles (i.e. 0.5 % wt and 1%wt) produces an increment of power consumption at given agitation speeds.

The relevant total power dissipation was translated into power number ( $N_p$ ) values, defined as

$$N_p = \frac{P}{\rho_L N^3 D^5} \quad (2)$$

where P is agitation power,  $\rho_L$  is liquid density, N is agitation speed ( $s^{-1}$ ) and D is the diameter of the radial bottom turbine ( $D = 0.095$  m). The results are also reported in Fig.4, where it can be observed that power number slightly decreases when increasing Reynolds

number, both in the presence or in the absence of solid particles, with average values ranging from 4.5 in the absence of solid particles to 5.5 with a 1% wt amount of solid particles.

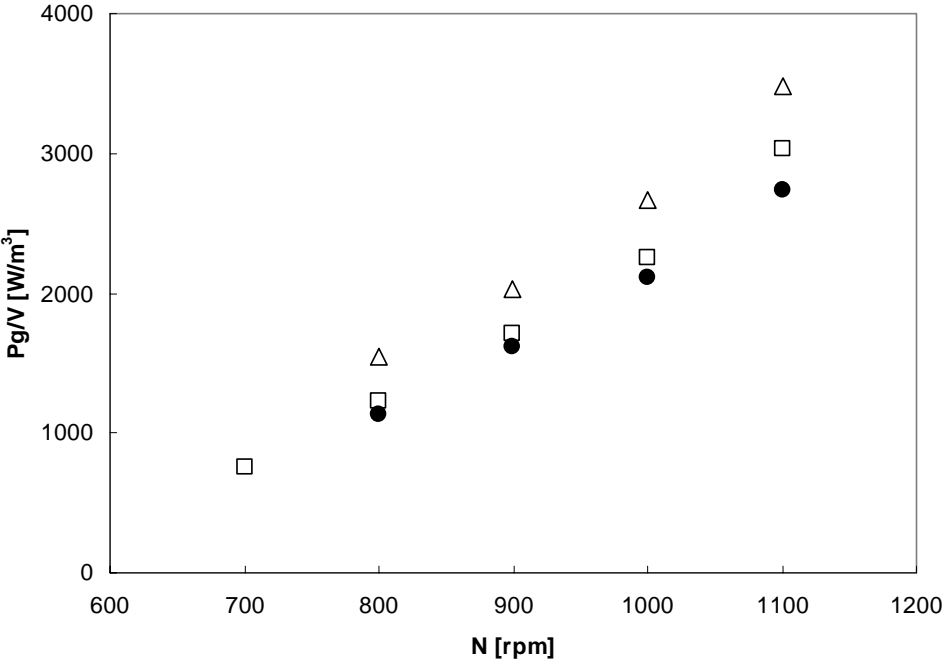


Figure 3. Specific power input versus agitation speed for various particle mass fractions: solid symbols, particle mass fraction=0 wt%; open squares, 0.5 wt%; open triangles, 1 wt%

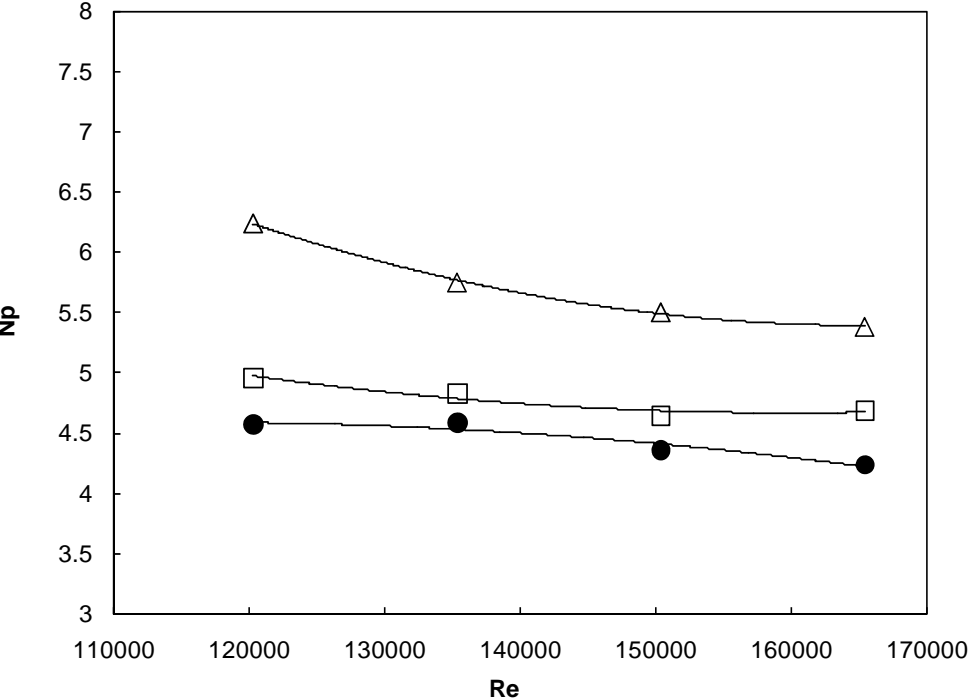


Figure 4. Experimental power numbers versus Re number for various particle mass fractions: solid symbols, particle mass fraction=0 wt%; open squares, 0.5 wt%; open triangles, 1 wt%

The reason behind the slight decrease is clearly the larger gas hold-up at higher rotational speeds. The relatively constant value observed may anyway be regarded as an indication of hydrodynamic robustness.

It is worth noting that the increase of power consumption due to the presence of just 1 % wt of solid particles is surprisingly high, being of the order of 25% . In order to explain such a large sensitivity of power on particles presence one may postulate that particles concentrate in the annular section and give rise to a sort of fluid friction increase that reduces fluid recirculation. This in turn lowers gas down-flow in the draft tube, hence increasing the power number of the lowest radial turbine. Considering that the lowest radial turbine having the largest diameter is the one that contributes the most to power consumption, the above chain of effects might well explain the observed dramatic increase of this last.

### 3.4 Mass transfer coefficient $k_{La}$

The  $k_{La}$  data obtained with the simplified dynamic pressure method described in Section 2 are reported in Fig. 5 versus specific power input for various particle mass fractions.

As it can be seen, the presence of solids reduces the mass transfer coefficient, although the differences between 0.5 and 1% of solid mass fraction are not large. Similar results were found also by Conway et al. [3] in their AGR reactor.

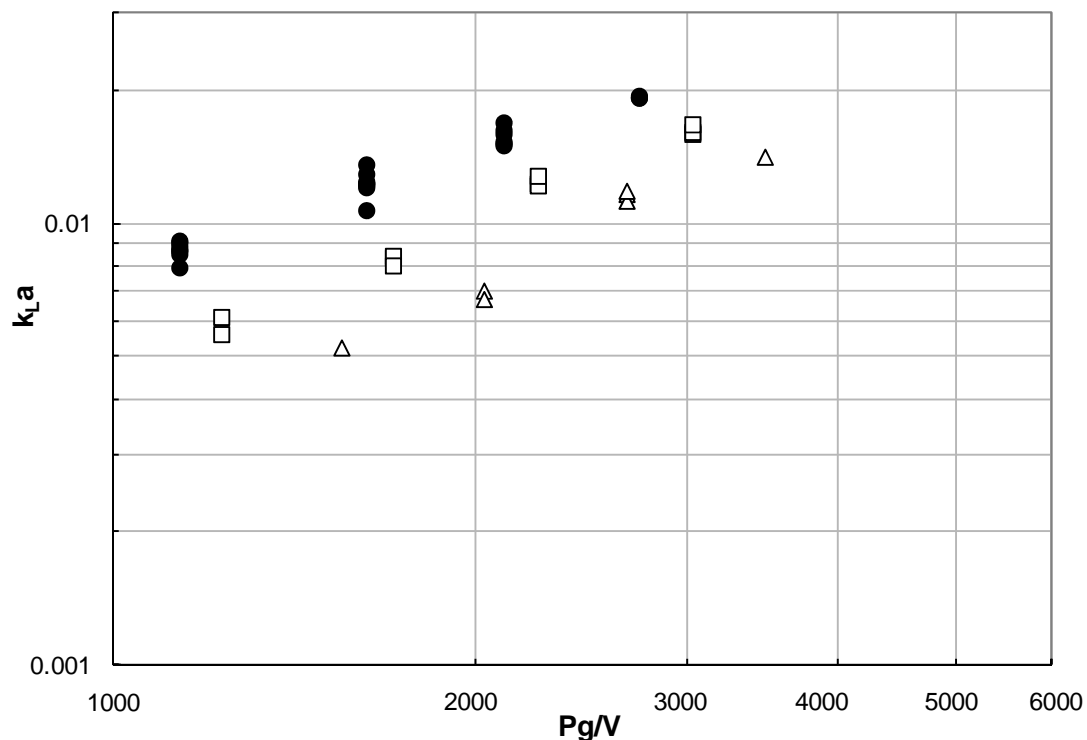


Figure 5. Mass Transfer Coefficient  $k_{La}$  versus specific power input for various particle mass fractions: solid symbols, particle mass fraction=0 wt%; open squares, 0.5 wt%; open triangles, 1 wt%

As explained in the discussion of the gas holdup, the presence of solids which accumulate in the annulus strongly modifies the gas hold up distribution inside the reactor 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 by the presence of solids in a vortex ingesting gas–liquid–solid reactor. A secondary effect may be that of turbulence damping by the particles [6] resulting in lower values of  $k_L$ .

The  $k_L a$  data are correlated with the specific power input by a power law of the form:

$$k_L a = \text{const} * \left( \frac{P}{V} \right)^n \quad (3)$$

With an average exponent  $n$  of about 1.1 for the three cases analysed.

#### 4. CONCLUSIONS

This preliminary study reports on the operation of the Long Draft Tube Self-ingesting Reactor (LDTSR) as a three-phase (gas–liquid–solid) reactor. In analogy with other self-ingesting reactors such as the AGR, it is found that the LDTSR is fairly suited for solids suspension, since the lowest radial impeller is very close to tank bottom and therefore particles are easily picked up and moved into the annulus. Moreover, large or heavy enough particles are not transported up to the draft tube brim and therefore are not recirculated through the impellers system, thus avoiding impeller impact attrition. Other designs, such as the gas-inducing impeller, admittedly have trouble in simultaneously suspending solids and ingesting a gas flow at reasonable specific power inputs. A detrimental effect of solids on the minimum speed for gas ingestion, gas holdup distribution and mass transfer coefficient is however observed. It can be stated that, generally, the presence of solids makes gas–liquid operation more difficult.

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