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Fluid Dynamics and Transport Phenomena

Micromixing characteristics in a gas–liquid–solid stirred tank with settling particles ☆

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ABSTRACT

The parallel-competing iodide–iodate reaction scheme was used to study the micromixing performance in a multi-phase stirred tank of 0.3 m diameter. The impeller combination consisted of a half elliptical blade disk turbine below two down-pimping wide-blade hydrofoils, identified as HEDT + $2WH_D$. Nitrogen and glass beads of 100 μm diameter and density 2500 $kg \cdot m^{-3}$ were used as the dispersed phases. The micromixing could be improved by sparging gas because of its additional potential energy. Also, micromixing could be improved by the solid particles with high kinetic energy near the impeller tip. In a gas–solid–liquid system, the gas–liquid film vibration with damping, due to the frequent collisions between the bubbles and particles, led to the decrease of the turbulence level in the liquid and caused eventually the deterioration of the micromixing. A Damping Film Dissipation model is formulated to shed light on the above micromixing performances. At last, the micromixing time t_m according to the incorporation model varied from 1.9 ms to 6.7 ms in our experiments.

1. Introduction

In a stirred reactor, the mixing process can be characterized on three scales: macromixing, mesomixing and micromixing. Micromixing is mixing at molecular scale, which may influence the selectivity, yield and quality of the final products. Therefore, the study of micromixing gives important guidance for industrial applications. The iodide–iodate technique, developed by Fournier *et al.* [1] and Guichardo *et al.* [2,3], has been extensively used for the micromixing study in a stirred tank, especially for single–phase, like Assirelli *et al.* [4–6] and Nouri *et al.* [7]. Moreover, the successive acid injection based on the iodide–iodate technique is really an advantage for the characterization of large vessels by reducing preparation time and experimental cost [2,4]. Meanwhile researchers developed different micromixing models, among which the incorporation model is the most extensively used one [8–10].

The literature related to the micromixing for multi-phase systems is limited especially for the gas–liquid–solid system. For gas–liquid systems, Lin and Lee [11] investigated the micromixing efficiency in a 1 L stirred tank sparged at 1.5, 10 and 20 vvm [vvm: (air volume/culture volume)/min]. They found that near the impeller tip, regardless of flooding, the agitation by bulk gas column or dispersed tiny bubbles could provide an efficient micromixed environment. In regions far from the tip, however, the occurrence of flooding could severely retard the micromixing. Brilman *et al.* [12] suggested that the effect of sparging on the product distribution was limited based on their research in a tank of 0.082 m diameter with V_S up to 0.028 $m \cdot s^{-1}$. Hofinger *et al.* [13] investigated the effects of gas on micromixing in a tank of 0.288 m diameter. In their semi-batch experiments with the test reagents fed near the impeller, the micromixing efficiency was not affected by sparging up to 1.5 vvm. However, a significant improvement of micromixing performance was observed by sparging gas when the reagents were fed near the top surface.

For solid–liquid systems, Barresi [14] showed that with the same stirring speed, the turbulence level was increased by the solid particles (150–212 μm; $\rho = 2500\ kg \cdot m^{-3}$) with a Rushton turbine but decreased with a pitched blade turbine. By PIV measurements, Montante *et al.* [15] suggested that moderate dampening of liquid turbulent fluctuations was found with particles of 115 μm diameter, while turbulence enhancement was observed with the 774 μm particles. Hofinger *et al.* [13] suggested that with 500 μm glass beads at mass concentrations up to 2.5 %, the micromixing was unaffected near the impeller and near the surface. At 11.63 % (by mass) when cloud formation was observed, the micromixing became significantly worse at both positions.

In general, the mechanisms by which the presence of bubbles and solid particles affect the turbulence level in the liquid phase are still unclear. Therefore, the successive acid injection of the iodide–iodate method was used to investigate the micromixing in multi-phase stirred tank. Firstly, the validity of the successive acid injection was tested. Then, the effects of gas flow rates Q_g, solid concentrations C_v and feed positions on the micromixing performance were systematically investigated. Finally, the micromixing time t_m and local specific energy dissipation rates ε_T according to the incorporation model were discussed.

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2. Experimental Setup

All the experiments were carried out in a dished-bottom cylindrical tank with internal diameter $T = 0.30$ m and a filled aspect ratio $H/T = 1.8$, as sketched in Fig. 1. The total liquid volume was 0.035 m^3. As a standard configuration, four 0.03 m wide baffles were symmetrically mounted on the wall. A half elliptical blade disk turbine [HEDT, Fig. 2(a)] was located at 0.4 T above the tank bottom, and two down–pumping wide–blade hydrofoils [WH_D, Fig. 2(b)] were mounted above it. The spacing between impellers was 0.48 T. The diameter D of all three impellers equaled to 0.33 T. The power number N_p of this impeller combination is 4.1 in single liquid phase. A ring sparger of diameter 0.8D with 27 symmetrical 0.002 m diameter downward-directed holes was located 0.33 T above the tank bottom. Two feed positions were chosen, as shown in Fig. 1. Both positions were located the same height as the bottom turbine; feed position 1 (P1) was 10 mm away from the impeller tip while feed position 2 (P2) was 30 mm away from the tank wall.

As the oxygen from air or water could oxidize the iodide to iodine, nitrogen and deionized water were used as gas and liquid phases respectively. The solid particles were 100 μm mean diameter glass beads of density 2500 $kg \cdot m^{-3}$. The gas flow rate Q_g ranged from 1.4 to 4.2 vvm, while the volumetric solid concentrations C_v ranged from 3 % to 9 %. The mean specific energy dissipation rate P_m was based on $W \cdot kg^{-1}$ of total mass in the tank including liquid and solid particles. The relationship between stirring speed and the torque was determined firstly for matching P_m with various gassing flow rates and particle concentrations, and P_m ranged from 1.2 to 2.8 $W \cdot kg^{-1}$.

3. Test Reaction

The iodide–iodate method was used in this work, as described by the following scheme:

$$H_2BO_3^- + H^+ \rightarrow H_3BO_3, \quad (1)$$

$$5I^- + IO_3^- + 6H^+ \rightarrow 3I_2 + 3H_2O. \quad (2)$$

The neutralization reaction (1) is considered instantaneous with H_3BO_3 as the desired product. The oxidation reaction (2) is fast but relatively slower than reaction (1) with iodine as the undesired product. The iodine forms further triiodide with iodide according to the quasi-instantaneous equilibrium:

$$I^- + I_2 \Leftrightarrow I_3^-, \quad (3)$$

$$K_B = \frac{[I_3^-]}{[I_2][I^-]}, \quad (4)$$

where K_B is the equilibrium constant of reaction (3). K_B is a function of temperature T' [16]:

$$\log_{10} K_B = \frac{555}{T'} + 7.355 - 2.575 \log_{10} T'. \quad (5)$$

During the experiments, a quantity of sulfuric acid was added into the mixture of KI, KIO_3 and $H_2BO_3^-$ at a specific feed position. When the micromixing conditions were perfect, H^+ was instantaneously dispersed and consumed by the first instantaneous reaction. However, when micromixing is not perfect, the local aggregation with high H^+ concentration would allow the slower reaction (2) more chance to take place.

The I_3^- concentration can be accurately measured by spectrophotometry at 353 nm [1]. According to the Beer–Lambert Law, the optical density D_0 is proportional to the concentration of I_3^- through the molar extinction coefficient ε of I_3^- at 353 nm:

$$[I_3^-] = \frac{D_0}{\varepsilon L}, \quad (6)$$

where L is the optical path length equal to 1 cm, and the molar extinction coefficient ε of I_3^- at 353 nm equals to 2535 $m^2 \cdot mol^{-1}$.

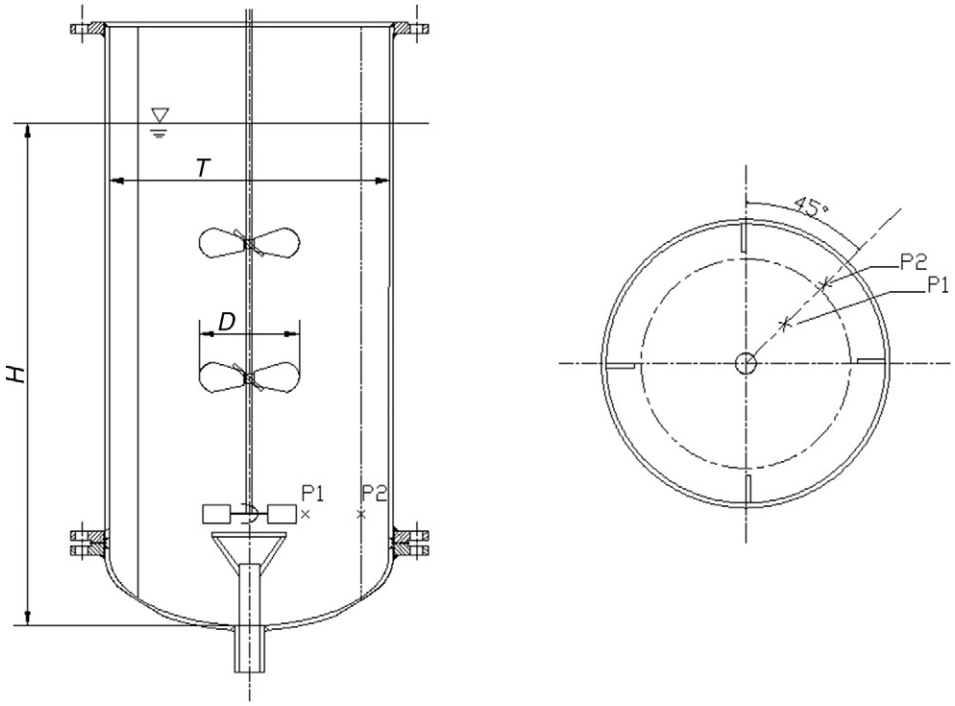

Fig. 1. Experimental setup and feed positions.