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Chemical Engineering Science 59 (2004) 4967–4974

Chemical Engineering Science

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Residence times and mixing of a novel continuous oscillatory flow screening reactor

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Received 28 February 2004
Available online 28 October 2004

Abstract

This paper is concerned with the fluid mechanics and mixing performance of a novel oscillatory flow screening reactor. Using fibre optic probes, a mixing coefficient k_m is determined for the system as a function of the applied fluid oscillation frequency and amplitude. In a continuous operation mean residence time and a backmixing coefficient g are estimated as a function of the oscillation conditions. Finally, in order to compare data with numerical simulations steady state flow data are also included.

The screening reactor presented an intermediate mixing behaviour throughout all the studied range of oscillation amplitudes (0–3 mm centre-to-peak) and frequencies (0–20 Hz). The backmixing was found to be highly dependent of the oscillation frequency and amplitude. Nevertheless, a stronger effect of the oscillation amplitude over the axial dispersion was detected presumably due to the increase of the mixing length. On the other hand, the increase of the oscillation frequency was concluded to have the increase in the radial mixing rates as the main effect. Thus, it was possible to achieve a decrease in the axial dispersion with the screening reactor using oscillatory flow, when compared to the laminar steady flow in a plain tube with the same mean internal diameter.

Keywords: Screening reactor; Oscillatory; Fluid mechanics; Dispersion; Mixing; Simulation

1. Introduction

A novel continuous screening reactor based on oscillatory flow technology (Harvey et al., 2001) has been recently presented by Harvey et al. (2003) as a new small-scale technology for reaction engineering and particle suspension applications. Enhanced performance in fluid micro-mixing and suspension of catalyst beads have been obtained with such a reactor. Due to its small volume (about 4.5 ml) this reactor is envisaged for applications in specialist chemical manufacture and high throughput screening, being also suitable for multiphase applications on a small-scale in the bioengineering field (e.g. fast parallel bio-processing tasks).

The main objective of the present work is to demonstrate the beneficial effect of the oscillatory flow in the screening reactor in terms of the mixing and residence time distribution of the liquid phase.

Tracing techniques are widely used for characterising the mixing of a liquid, a solid or a gas phase (Boyer et al., 2002). For continuous processing devices, the tracer injection and response method have been widely used to study mixing characteristics of reactors. Techniques such as the moment, weighted moment, Laplace and Fourier transform domain analysis and time domain analysis have been employed to analyse the tracer response data.

Time domain analysis of residence time distribution (RTD) of the liquid phase in conventional oscillatory flow reactors (OFRs) has been reported by several authors (e.g. Mackley and Ni, 1991, 1993) using conventional tracer techniques (e.g. conductivity measurement). More recently

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doi:10.1016/j.ces.2004.09.013

Fitch and Ni (2003) applied a non-intrusive state-of-the-art laser-induced fluorescence technique to study the RTD of the liquid phase in conventional OFRs and concluded that intrusive experiments using conductivity probes can have problems of mass transfer into the membranes of the probes, leading to misleading results.

In this study, the mixing and residence time distribution of the liquid phase in a novel screening reactor is determined using the conventional unsteady tracer injection but eliminating some of it pitfalls by applying fibre optic probes. In the interpretation of RTD curves flow the reactor's behaviour was tested by two models that usually represent intermediate mixing behaviours; the differential and the stagewise backmixing model (Mecklenburgh and Hartland, 1976). Model parameters were determined by fitting the Laplace transform of these models with that of the experimental pulse response (tracer concentration along the time).

2. Materials and methods

The Screening reactor: It is formed by several jacketed glass tubes, with 4.4 mm internal diameter and 35 cm length. Each tube has a volume of ca. 4.5 ml and is provided with smooth periodic constrictions (SPCs) (Fig. 1a). Fluid oscillations were achieved by a rotative ceramic piston pump (CKCRH0, Fluid Metering Inc., New York, USA), working in a close-loop, connected to the bottom of the reactor. Good sinusoidal fluid oscillations may be obtained. The control of oscillation amplitude (from 0 to 3 mm) was made by turning an easy-grip flow control ring in the pump head. This work was focused in a range of oscillation frequencies from 0 to 20 Hz, which was controlled by the rotation speed of the ceramic piston. In all, a precise control of both oscillation amplitude and frequency was obtained. In this work, all values of amplitude are expressed centre-to-peak. All experiments were performed at room temperature (20 °C) and using distilled water as main fluid.

Liquid phase studies: The coloured tracer used for mixing and RTD studies of the liquid phase was an aqueous solution of Indigo carmine obtained from Merck (Darmstadt, Germany). Continuous and batch experiments were performed leading, respectively, to the determination of mixing times and RTDs, obtained from the concentration versus time curves (*C*-curves).

In continuous operation, one SPC tube was fixed vertically and mounted according to Fig. 1(b). A continuous liquid flow rate was coupled using a peristaltic pump. Experiments were prepared by continuously injecting an aqueous solution of 0.2 kg/m^3 of tracer in the SPC tube. Once the concentration was stable, tracer injection was stopped and replaced by clean water (the starting point of the experiments, i.e., $\theta = 0$). The water net flow was maintained until the concentration of tracer was null at the exit. A transmission dip optical micro-probe (FDP-UV-micro-1, Avantes, Eerbeek, The Netherlands) was used for on-line monitoring of the tracer concentration at the inlet (first cavity—near the injection) of the SPC tube. At the outlet, tracer concentration was attained by a reflection probe (FCR-7UV200-1,5×100-2, Avantes, Eerbeek, The Netherlands). The two probes were at a distance of 343 mm. Monitoring of concentrations along the experimental time led to *C*-curves. Further experiments with dissimilar flow rates were performed to determine the effect of flow rate over the RTD during continuous operation.

At the batch operation mode, one SPC tube was fixed vertically and 0.2 ml of the tracer with a concentration of 0.5 kg/m^3 was injected at the top of the reactor. Operational volume was about 4.5 ml. Fluid oscillation was started (at time $t = 0$) and the tracer concentration inside the SPC tube was on-line monitored at the top cavity (in batch mode, it is the same cavity where injection was performed) using the transmission dip optical micro-probe until a steady (constant) value was attained. The time required to achieve a specified level of uniformity, U, where $0 < U < 1$ (see Eq. (3) for definition), was defined as the mixing time, t_U.

In both operational modes (batch and continuous), the optical probes were connected to a multi-channel optic spectrometer (AvaLight-2048, Avantes, Eerbeek, The Netherlands). Readings were made at each $\frac{1}{9}$th of a second and each three consecutive readings were averaged resulting in experimental points available at each $\frac{1}{3}$rd of a second. The response of the fibre-optical system was highly linear in the entire range of tracer concentrations.

Numerical simulations of RTD: Further studies using the computational fluid dynamics (CFD) technique were performed where an accurate prediction of the distribution of residence times was sought. It has been previously reported by Harvey et al. (2003) that a 2D-axisymmetric model may show a good agreement of simulated flow patterns at smooth oscillation conditions (low oscillation amplitudes). For further details about the CFD model please refer to Harvey et al. (2003). In this study, the ability of such a model to predict the RTD of the liquid phase was tested at steady flow and different continuous flow rates, using Fluent software (Fluent Inc, New York, USA).

Parameter estimation from non-ideal flow models: Axial dispersion was estimated by comparison of the numerical Laplace transforms of selected hydrodynamic models with that of the pulse response, around a point T_{ref} that suppresses the effect of the exponential term (tail in the *C*-curve). At Laplace's domain, the response of a system can be represented by the inlet times a transfer function, $g(T)$, as follows:

$$\bar{x} = \bar{x}_{\text{in}} g(T). \tag{1}$$

In terms of inlet, instead of considering $\bar{x}_{\text{in}}$ (the input in Laplace's domain) for a negative step, all experimental concentrations x were converted to $[x_{\text{in}} - x]$, where x_{in} is the tracer concentration in the inlet stream. This procedure transforms the negative step response into a positive step response significantly simplifying all the calculations.

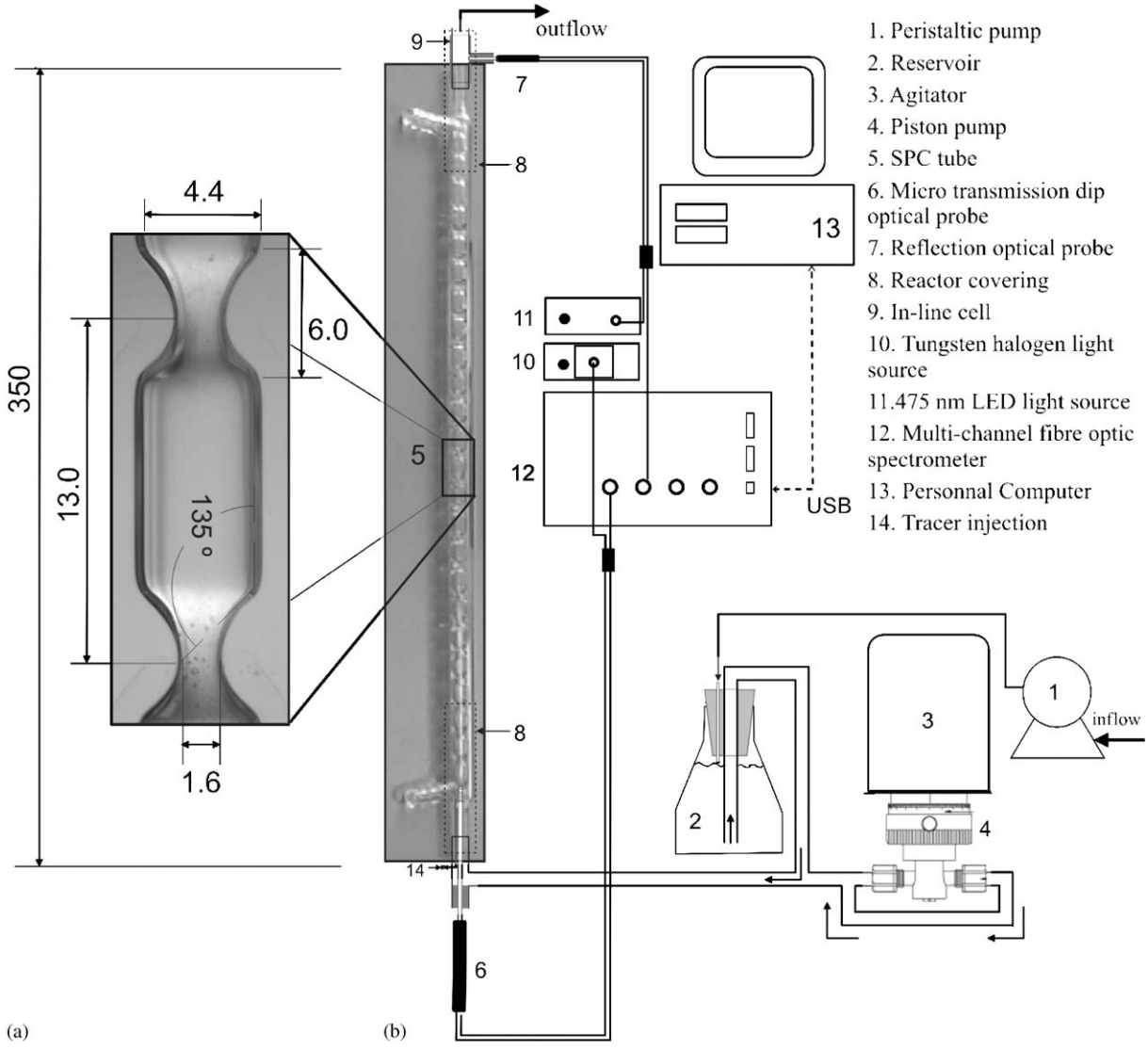

Fig. 1. (a) Geometry of a SPC tube (units in mm), (b) example of the experimental apparatus used in the study of RTD of the liquid phase.

In this work, axial dispersion was fitted to two hydrodynamic models. The differential backmixing model (Mecklenburgh and Hartland, 1976) (Fig. 2a) considers the backmixing as an infinite backflow with infinite transverse mixing of similar order of magnitude and usually applies to differential equipment such as pipes and tubular reactors. An element of fluid can move backwards and forwards, and relative to the conventional flow it has an equal chance of moving in either direction. The stagewise backmixing model (Mecklenburgh and Hartland, 1976) (Fig. 2b) refers to backflow of fluid between perfectly mixed stages in a direction opposite to the main flow (Sleicher, 1960). The backmixing is quantified by a coefficient, g, which is defined as the ratio of backwards flow between stages to the net forward convective flow. This model assumes that all units are connected by zero volume sections. For the present case study, the number of stages was 26, which corresponds to the average number of cavities in a single SPC tube. Transfer functions for both differential and stagewise backmixing models can be found in Mecklenburgh and Hartland (1976).

3. Results and discussion

Determination of a batch mixing time as a function of the oscillation conditions: Two hundred different experiments were carried out in batch mode and at different oscillation conditions. From the monitored concentration along the experimental time (Fig. 3a) a mixing time parameter, t_U, was obtained. It is defined as the time to achieve a certain level of uniformity, U (usually 90%, 95% or 99%). However, a different approach allowed correlating t_U with U and an empirical mixing coefficient, $k_m[\mathrm{T}^{-1}]$. A mass balance performed to the tracer concentration x in the cavity of the bottom of

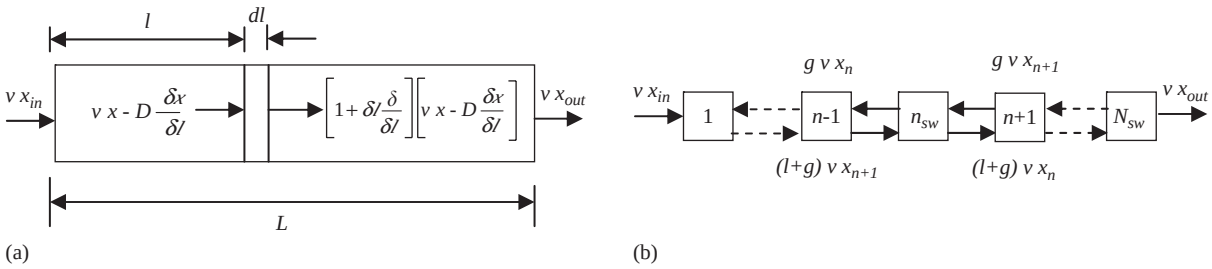

Fig. 2. Representation of flow in (a) a differential and (b) a stagewise reactor with backmixing (the mass balance is only presented for the stage n) (Mecklenburgh and Hartland, 1976).

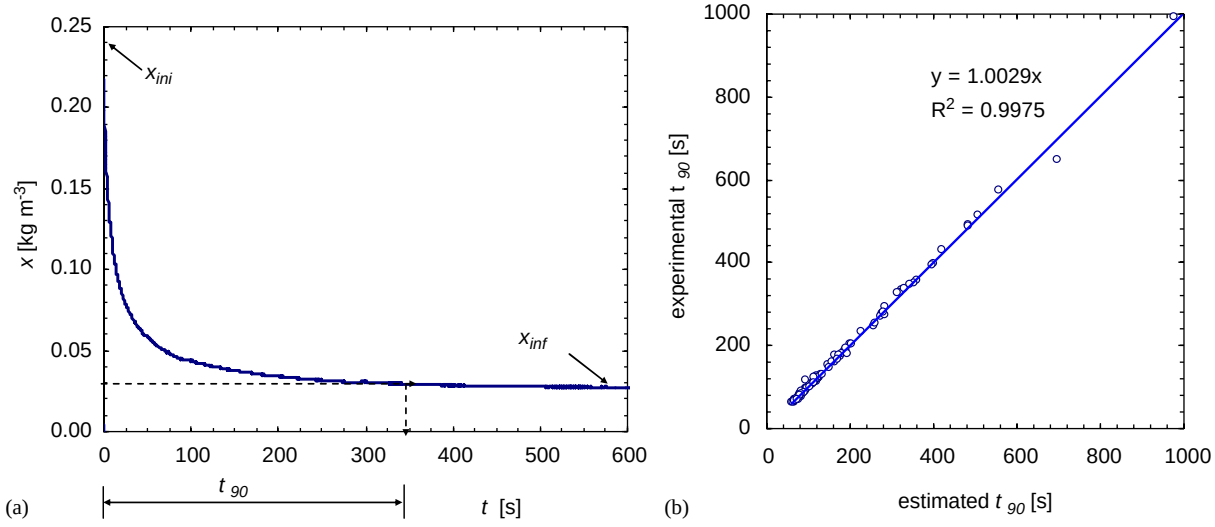

Fig. 3. (a) Determination of mixing time t_{90} parameter from experimental data at 20 Hz and 1 mm, (b) comparison of experimental t_{90} parameter with estimated values with Eq. (5).

the tube (the cavity which is at farthest distance from the injection point) yields, after integration along the experiment time t

$$\ln\left(\frac{1/x_{\text{inf}} - 1/x_i}{1/x_{\text{inf}} - 1/x_{\text{ini}}}\right) = -k_m t, \tag{2}$$

where x_{inf} is the concentration at infinite time, x_{ini} is the initial concentration and x_i is tracer concentration at time t_i. Eq. (2) allows the determination of k_m directly from the experimental data. The main advantage of this procedure is that mixing times are not limited to defined levels of uniformity but to a wide range of U. Eq. (2) works very well for $0.5 < U < 0.99$, with correlation coefficients, R, above 0.99 (results not shown here).

As for the cavity at the top (where x_i was monitored) U is given by

$$U = \frac{x_{\text{inf}}}{x_i} \tag{3}$$

it can be demonstrated that the general equation for t_U is

$$t_U = -\frac{1}{k_m} \ln\left[\frac{1-U}{1 - x_{\text{inf}}/x_{\text{ini}}}\right]. \tag{4}$$

When x_{inf} is very small when compared with x_{ini} (the most usual situation), the logarithm term simplifies to $\ln(1-U)$.

Fig. 4 presents the variation of k_m with x_0 and f in the studied range. It is clear that both x_0 and f affect k_m and thus the mixing time in a non-linear way. The following dependency of t_U (in seconds) was obtained from experimental data fitting:

$$t_U = \frac{1}{0.0072 \ln(x_0^{2.35} f/2.75)} \ln[1-U]. \tag{5}$$

This equation is valid only for values satisfying $x_0^{2.35} \cdot f > 2.75$ and are restricted to the SPC geometry. Otherwise, Eq. (5) cannot be applied and data from batch experiments (Fig. 3) suggest that a near-null axial dispersion is reached, thus approaching a plug flow behaviour.

The comparison of parameter t_{90} ($U = 90\%$) estimated from Eq. (5) with experimental values led to a maximum error of 5.7% in all studied range of oscillation frequencies and amplitudes (0–20 Hz and 0–3 mm, respectively), showing a very good fit (Fig. 3b).

The mixing time correlation given by Eq. (5) shows that k_m is more sensitive to x_0 than f. Presumably, this relates

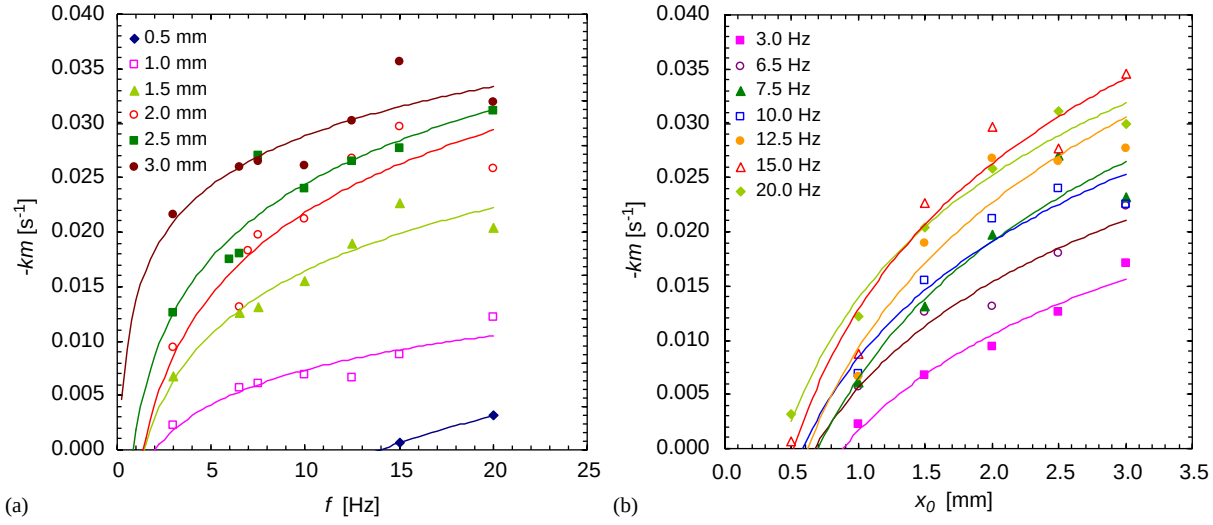

Fig. 4. Variation of the mean values of mixing coefficient k_m with fluid oscillation (a) frequency and (b) amplitude at different oscillation conditions.

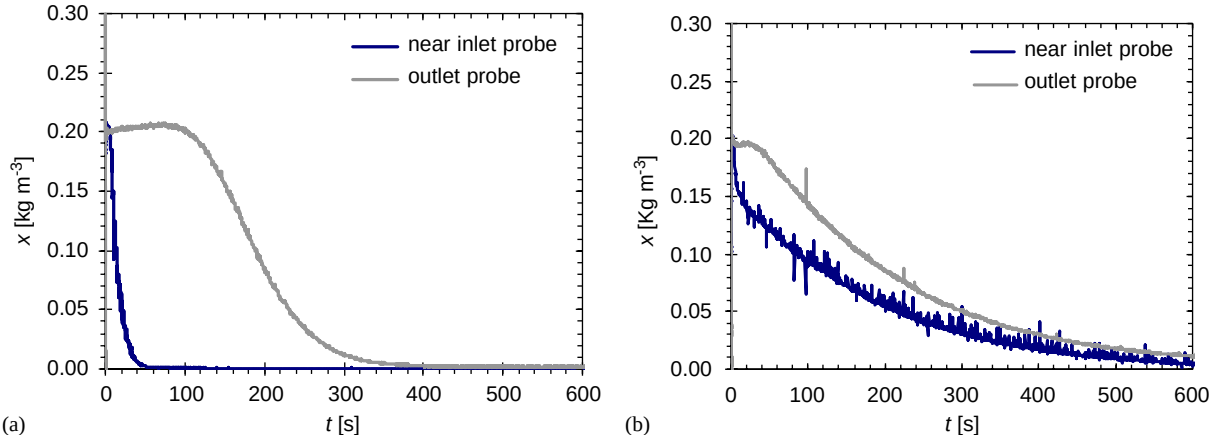

Fig. 5. Experimental *C*-curves (tracer concentration along experimental time) at a point near the inlet and at the outlet of a SPC tube at: (a) 3 Hz and 0.5 mm; (b) 20 Hz and 3.0 mm. Net flow rate of 1.94 ml/min.

with the effect that x_0 has on the nature of the eddies formed and thus on dispersion along the tube. This confirms PIV observations and CFD simulations previously reported in Harvey et al. (2003). The increase in f leads essentially to an increase in mixing intensity inside each cavity and thus not necessarily to an increase in the axial dispersion. This allows to conclude that in the screening reactor: (i) the increase in x_0 (at constant f) leads essentially to an increase in the mixing length (thus increasing the axial dispersion) and (ii) the increase in f (at constant x_0) leads essentially to an increase in the radial mixing. The second conclusion can only be confirmed by RTD analysis (see next section).

Liquid phase RTD determination for continuous flow as a function of the oscillation conditions: About 100 different experiments were performed in the continuous mode, at different oscillation conditions and at a constant liquid flow rate of 1.94 ml/min. From the monitored tracer concentration at the outlet and near the inlet of the tube along the experimental time (*C*-curves) (see examples in Fig. 5) the mean residence time, $\bar{t}$, was then calculated. Since discrete points were available, the mean residence time of the tracer was determined as follows:

$$\bar{t} \cong \frac{\sum_{i=1}^{\infty}\{1/2[t_i + t_{i-1}][x_i - x_{i-1}]\}}{\sum_{i=1}^{\infty}\{x_i - x_{i-1}\}}. \quad (6)$$

The obtained average mean residence times of the tracer are represented in Fig. 6. Apparently, the mean residence time is not affected by the oscillation conditions and, for the flow rate used, it was determined to be close to 200 s (using Eq. (6)). However, the flow rate used corresponds to an hydraulic time of ca. 135 s, which is about 32.5% lower than the average mean residence time determined for the tracer. This demonstrates that the tracer is effectively lagged

in the cavities, within vortex rings reported by Harvey et al. (2003).

Estimation of values of Peclet P or backmixing g for both backmixing models considered in this study was performed by fitting the experimental transfer function ($g_{out}(T)$) to the (discretised) theoretical equations (Mecklenburgh and Hartland, 1976), assuming a perfect step input at the inlet. The minimum square function in the range $[0.5.T_{ref}–2.T_{ref}]$ (around a value of T_{ref} equal to 2) was considered to be the best fitting. The perfect step input assumption was confirmed by comparison of the estimated parameters with those obtained considering an imperfect step at the inlet (results not shown).

Values of g estimated according to the stagewise backmixing model are presented in Fig. 7, considering 26 stages (the same as the number of cavities). Differential and stagewise backmixing models are inter-convertible and so only parameter g is presented here. The theoretical relation between the values of Peclet number, P (differential backmixing model) and backmixing, g (stagewise backmixing model) (Mecklenburgh and Hartland, 1976) was confirmed to be $g + 0.5 = N_{sw}/P$, with a cross correlation coefficient, R, equal to 0.9993, while using parameters fitted assuming a perfect step input.

Usually, values of P above 1 indicate a behaviour near that of a CSTR, while values of P above 50 indicate a behaviour near that of a plug flow reactor (Mecklenburgh and Hartland, 1976), corresponding to values of g of 25.5 and 0.02, respectively, in the case of one SPC tube. In fact, an intermediate behaviour was obtained in a single SPC tube throughout the studied range of oscillation amplitudes and frequencies.

It was difficult to draw a general conclusion from Fig. 7, but it was clear that both x_0 and f affect the backmixing (or axial dispersion). As previously concluded from mixing times analysis, it was also possible to conclude from analysis of RTD that an increase in f does not affect the dispersion on the same order of magnitude as an increase in x_0 does. Moreover, is was found that axial dispersion can effectively be decreased at any constant x_0 (from 0 to 3 mm) by operating at a f of 10 Hz (see Fig. 7b). The best oscillation conditions for a near-plug flow behaviour were found to be 0.5–1.0 mm and 7.5–10 Hz (see Fig. 7a, b), while the near-completely mixed state can be accomplished at high oscillation amplitudes (> 3 mm) and frequencies (> 20 Hz), due to applied high backflow rates.

In Fig. 7, the grey interrupted lines establish the limits for g: the right line corresponds to the (lower) observed limit for backmixing, while the left line corresponds to the (higher) theoretical average backflow (g_{teo}) imposed by the oscillatory flow. It can be demonstrated that g_{teo} is given by

$$g_{teo} = \frac{Re_0 \mu d}{4\rho v}. \quad (7)$$

Fig. 7 clearly shows that operation at low f (< 3 Hz) closely follows Eq. (7). Operation at high f leads to a lower

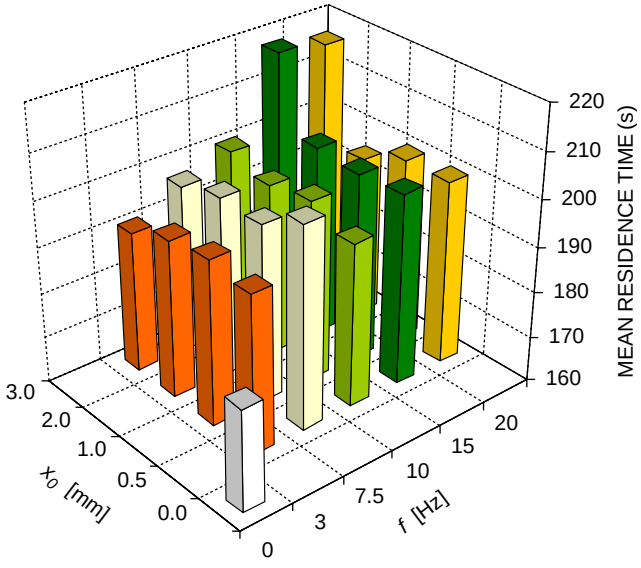

Fig. 6. Mean residence time of the tracer, measured at the outlet of the screening reactor for various oscillation amplitudes, x_0, and frequencies, f, at a fluid flow rate of 1.94 ml/min.

g for the same Re_0. Thus, low f denotes low radial mixing rates (and g approaches g_{teo}) while high f means increased radial mixing rates. However, high f also means increased axial velocities and thus a compromise must be considered between increased radial mixing and axial dispersion.

Starting from a non-oscillating state, a decrease in g was observed at Re_0 between 100 and 200 (Fig. 7). This can be explained by increased radial mixing, as reported by Howes et al. (1991) and is due to a break of flow symmetry (at a Re_0 of about 100). It confirms *PIV* visualisations reported by Harvey et al. (2003).

It has been possible to obtain a significant decrease in g (or axial dispersion) at a Re_0 below 400–500 in a single SPC tube when compared with the laminar flow in a tube with the same mean diameter and length (Danckwerts, 1953). This clearly shows that oscillatory flow can effectively reduce backmixing, when compared to the flow in a tube without oscillation.

Effect of liquid flow rate on RTD determination and comparison with numerical simulations for steady flow: The effect of the flow rate on fluid dispersion was studied by performing experiments in the absence of oscillations (Fig. 8). The mean residence times intended for future industrial applications are in the order of minutes to hours. For this novel screening reactor this means very low flow rates, namely below 1 ml/min. Previous CFD studies (Harvey et al., 2003) showed that flow separation (development of counter flow, leading to vortex rings) occurs in the screening reactor at a *Re* of about 10, i.e., for a flow rate of ca. 2 ml/min. Also according to previous studies on oscillatory flow (Harvey et al., 2001), the coupling of a net flow above that *Re* value will not significantly affect the fluid mechanics (or the RTD) behaviour of the screening reactor and so axial dispersion

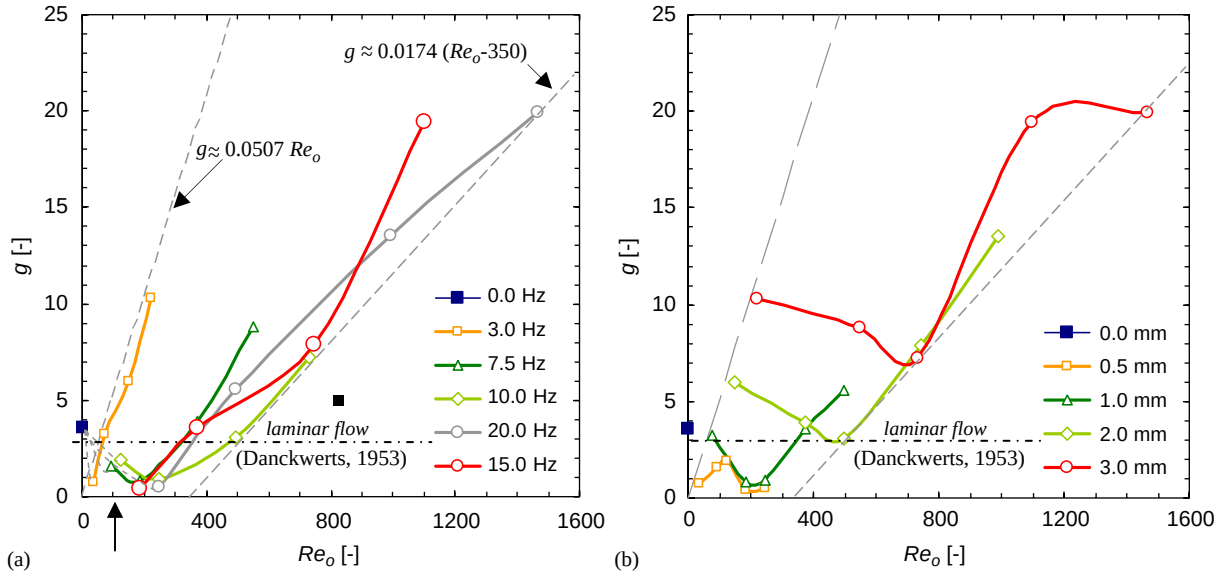

Fig. 7. Fitted backmixing parameter g using Laplace transforms as a function of Re_o at: (a) various x_0 and (b) various f. All values were obtained considering a perfect pulse input, a flow rate equal to 1.94 ml/min and a number of stages equal to 26.

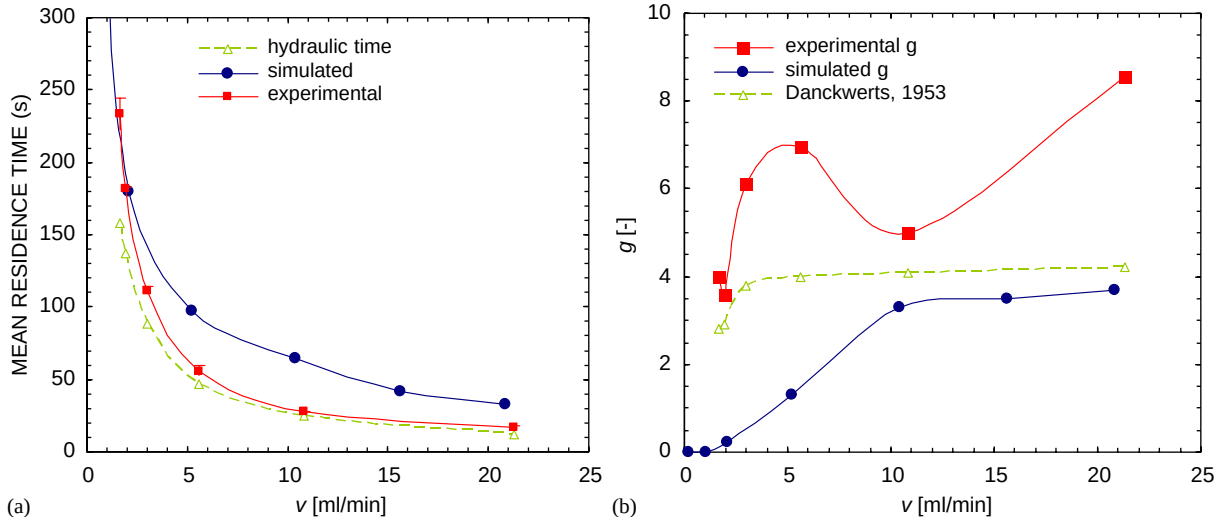

Fig. 8. Effect of net flow rate over (a) tracer mean residence time and (b) backmixing, g, assuming a perfect step input at steady flow (no fluid oscillations). A comparison is presented between experimental (■) and simulated values (●) using a 2D-axisymmetric model.

values found in this study are supposed to remain valid for lower flow rates. Note that this assumption it is only valid for values of axial dispersion, D, not to dimensionless parameters P or g since the last two are dependent on the flow rate.

Experimental values of g were compared with results from CFD simulations using a 2D-axisymmetric model. A comparison with laminar flow in an unbaffled tube (with the same mean internal diameter) is also shown (Danckwerts, 1953) in Fig. 8. Differences between simulated and experimental results (Fig. 8b) can be explained by differences in $\bar{t}$: knowing that g is a best fitted parameter based on the experimental dimensionless times ($\theta = t/\bar{t}$), differences between experimental and simulated $\bar{t}$ (Fig. 8a) consequently led to different best fitted g (Fig. 8b). In fact, simulations using a 2D-axisymmetric model tend to over-estimate the mean residence time of the tracer (Fig. 8a). This can be due to two different reasons: (1) the CFD model does not predict correctly the mixing process; (2) some tracer is lost through the symmetry axis boundary due to the nature of a 2D-axisymmetric model. In any case, the 2D-axisymmetry model has shown to be not suitable for RTD predictions. Strategies for matching RTD simulations may involve the optimisation of the time step or the use of a 3D-mesh (re-

quiring extended computation times). This will be the subject of future publications.

Experimental backmixing was found to be higher than the backmixing parameter fitted to a *C*-curve of a laminar flow in a tube in the absence of oscillations. This means that the existence of constrictions contributes to an increase in the axial dispersion but such increase can globally be overcome when imposing the oscillatory flow in the screening reactor, as demonstrated by RTD of the liquid phase (Fig. 7).

4. Conclusions

Experimental data presented in this paper characterise certain aspects of a novel screening reactor based on oscillatory flow. In particular, the batch mixing data presented in terms of a mixing coefficient k_m has shown, as anticipated, that mixing time depends on both oscillation amplitude and frequency and that oscillation amplitude has a stronger effect than frequency.

In terms of experiments at continuous flow both mean residence time and backmixing coefficient g were found to be sensitive to the oscillatory flow conditions and presumably this is a consequence of the detached nature of the eddy mixing generation. The steady flow experiments reported in this paper offered the opportunity to compare experimental data with numerical simulations using Fluent software but an incomplete agreement was achieved. It was clearly demonstrated that oscillatory flow can positively decrease axial dispersion in baffled tubes, being possible to approach the residence time distribution of a perfect plug flow behaviour.

Notation

d	mean internal tube diameter, m
D	axial dispersion, $m^2\,s^{-1}$
f	oscillation frequency, s^{-1}
g	backflow ratio, dimensionless
k_m	mixing coefficient, s^{-1}
N_{sw}	total number of discrete tanks, dimensionless
P	Peclet number ($P = vL/D$), dimensionless
Re_0	oscillatory Reynolds number ($Re_0 = 2\pi f x_0 \rho d/\mu$), dimensionless
t; $\bar{t}$	time; mean residence time of the tracer, s
U	uniformity level ($U = 0$ to 1), dimensionless
v	volumetric flow rate, $m^3\,s^{-1}$
x	concentration, $kg\,m^{-3}$
x_0	oscillation amplitude, mm

Greek letters

θ	dimensionless time ($\theta = t/\bar{t}$)
μ	fluid viscosity, $kg\,m^{-1}\,s^{-1}$
ρ	density of the fluid, $kg\,m^{-3}$
τ	mean hydraulic time ($\tau = V/v$, being V the reactor volume), s^{-1}

Acknowledgements

Thanks are due to Fundação para a Ciência e a Tecnologia (FCT) by financial support of the co-author Nuno Reis by means of scholarship SFRH/BD/6954/2001.

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