



# Development and evaluation of novel designs of continuous mesoscale oscillatory baffled reactors

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## ABSTRACT

Oscillatory baffled reactors (OBRs) are a form of plug flow reactor, ideal for performing long reactions in continuous mode, as the mixing is independent of net flow rate leading to more compact and practical designs. Mesoscale OBRs are currently being developed for laboratory-scale processes. The systems are designed to scale-up to industrial scale directly, or to be used as small-scale production platforms in their own right. Three different meso-reactor baffle designs (integral baffles, helical baffles and axial circular baffles (or “central”) baffles) were developed. These designs were chosen, as they are easily fabricated at “mesoscales” (here typically ~5 mm diameter) and can be operated at low flow rates ( $\mu\text{l}/\text{min}$  to  $\text{ml}/\text{min}$ ), whereas conventional designs of OBRs cannot. It was found that an increase in the net flow Reynolds number increased the optimum range of oscillatory Reynolds numbers over which plug flow can be achieved. The oscillation conditions had little effect on the residence time distribution behaviour at net flow Reynolds numbers above 25. These designs are effective and robust in scaled-down production of high added value products and decreasing the reagents required.

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## 1. Introduction

Applying oscillatory flow brings advantages such as enhancing heat and mass transfer, mixing in flocculation, low shear and controllable mixing conditions [10,12,18]. Unlike tubular reactors where a high superficial velocity is required to obtain good mixing, OBRs can provide plug flow behaviour at net flow Reynolds numbers in the laminar flow regime, as the plug flow nature of the flow is produced by interaction between the oscillating fluid and the sharp-edge of baffles, which creates series of well-mixed volumes. This largely decouples achievement of plug flow from net flow velocity, leading to the OBR's niche application in converting long residence time batch processes to continuous, where conventional plug flow reactor designs have an impractically high length-to-diameter ratio [3].

The fluid mechanics inside continuous OBRs are governed by geometric parameters (baffle spacing and opened cross-sectional area) and dynamic parameters (expressed in the 3 dimensionless groups below). The oscillatory Reynolds number describes the intensity of mixing in the reactor, whilst the Strouhal number measures the effective eddy propagation in relation to the tube

geometry.

$$\text{Net flow Reynolds number : } Re_n = \frac{\rho Du}{\mu}$$

$$\text{Oscillatory Reynolds number : } Re_o = \frac{2\pi f x_o \rho D}{\mu}$$

$$\text{Strouhal number : } St = \frac{D}{4\pi x_o}$$

where  $f$  (Hz) is the frequency of oscillation;  $x_o$  (m) is the centre-to-peak amplitude of oscillation;  $u$  (m/s) is the superficial velocity of the liquid with its density  $\rho$  ( $\text{kg m}^{-3}$ ) and viscosity  $\mu$  ( $\text{m}^2 \text{s}^{-1}$ ) through a tube diameter  $D$  (m).

Characterisation of mixing behaviour in conventional OBRs has been extensively studied by evaluating the residence time distribution (RTD) of a tracer injection [1,4,5,8,11,15,17,19,20]. Dickens et al. [1] found that at a fixed  $Re_n$  of 110 and oscillation frequency of 3.5 Hz, the minimum axial dispersion coefficient was at a range of  $St$  of 0.6–1.7 and  $Re_o$  of 426–924. A comprehensive investigation of the effects of different net flows, oscillation conditions and their interdependence on the RTD performance in a 24 mm diameter, 2.8 m long conventional baffled tube was carried out [20]. The results showed that the system operated close to the plug flow at a velocity ratio of  $Re_o$  to  $Re_n$  between 2 and 4 and the RTD performance was less dependent on the oscillation amplitude when the required oscillatory Reynolds number was achieved.

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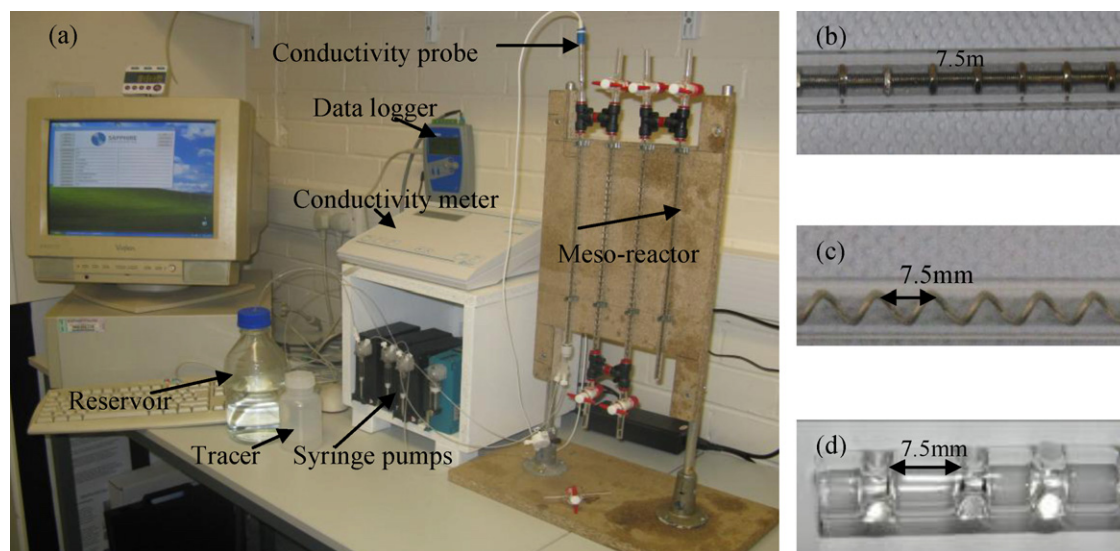


Fig. 1. (a) Experimental set-up for RTD; (b) helical baffles; (c) central baffles; and (d) integral baffles.

Small oscillatory baffled reactors (or “meso-reactors”, smooth periodic column “SPC”) have recently been developed and implemented [13,14,21]. They can be operated at low net flow Reynolds numbers, e.g. 10–35, which can be suitably applied in the fields of specialist chemical manufactures, high-throughput screening and bioengineering. A number of findings indicated that the conditions to obtain plug flow were slightly different to those in conventional designs of OBRs in terms of minimum superficial velocity and oscillatory conditions. The  $Re_0$  at which near plug flow performance is achieved, for instance, is between 10 and 100 for meso-reactors [2], but is above 100 for conventional OBRs [9]. A minimum  $Re_n$  to achieve convection was above 50 for the OBRs while it was above 10 for the SPC. Reis et al. [14] also found that a near plug flow behaviour was obtained at an amplitude of 0.5–1 mm and a frequency of 7.5–10 Hz at a fixed net flow rate of 1.94 ml/min or  $Re_n$  of 8.54.

Moreover, the fluid mechanics in the mesoscale reactors were found to be much more sensitive to oscillatory amplitude even though the required  $Re_0$  was fixed. For example, the structure of the eddies was axisymmetric at 4.1 Hz/1.0 mm ( $Re_0 = 117$ ) but non-axisymmetric at 1.1 Hz/3.8 mm ( $Re_0 = 116$ ) [2].

Although the design methodology has been developed for conventional OBRs [19], there is little research relating to meso-scale OBRs. In this work, the RTDs of three mesoscale oscillatory baffled reactors (namely central, integral and helical baffles) were investigated over a wide range of net flows from 1.0 to 8.0 ml/min, corresponding to values of  $Re_n$  from 4.3 to 34.0. The net flow of the fluid had oscillatory flow conditions ranging from 0.5 to 4 mm amplitude and 1–6 Hz frequency superimposed upon it. The effect of net flow, oscillatory flow and their interdependence on the fluid mixing inside the reactors was studied in order to evaluate a suitable condition for the three designs in which the plug flow can be approached.

## 2. Experimental and numerical methods

### 2.1. Experimental set-up

The experimental set-up for the RTD in the three mesoscale baffled reactors is shown in Fig. 1. A series of “Confluent PVM” syringe pumps (Eurodyne Ltd.) were used to provide a superficial flow, inject tracer and oscillate the fluid. Water was continuously

dispensed from a reservoir into the systems at flow rates ranging from 1.0 to 8.0 ml/min ( $Re_n = 4.3–34.0$ ). The process fluid was oscillated at various oscillation conditions, i.e. 0.5–4 mm amplitude and 1–6 Hz frequency. The frequency was controlled by adjusting the speed of the piston movement, whereas the amplitude (centre-to-peak) was varied by setting the amount of volume dispensed. These parameters were monitored via a computer.

Before starting an experiment, the system was completely full and air-free. The pumps were adjusted to a required superficial velocity and oscillation condition (frequency and amplitude). A known amount of the KCl (Sigma–Aldrich) tracer was injected into the system at a highest flow rate at the bottom of the column in order to obtain a sharp pulse injection. At the outlet, the corresponding conductivity of the tracer was measured using an E61M014 conductivity probe connected to a CDM210 conductivity meter (Hach–Lange Ltd.). The data was recorded until it reached zero and logged.

Three reactor tubes with central, helical and integral baffles are shown in Fig. 1(b)–(d) below. Baffles were inserted both in the tubes and the connecting sections to eliminate stagnation points. The geometry of the reactors was maintained similarly to that of conventional OBRs in terms of spacing of baffles (1.5 times the diameter of the tube) and open cross-sectional area (25–40%). The three types of baffles were hexagonal solid discs, helices and smooth periodic baffles. The 4 mm stainless steel solid discs were evenly spaced along a 2 mm diameter studding, giving an open cross-sectional area of 36% (Fig. 1(b)). The helical baffles’ pitch was 7.5 mm and the internal diameter was 2.6 mm, giving an opened cross-sectional area of 26% (Fig. 1(c)). For the case of the integral baffles, the smooth periodic baffles were equally spaced at 7.5 mm and the orifice diameter was 2.5 mm (Fig. 1(d)).

### 2.2. RTD analysis

RTDs can be quantified using statistical moment methods such as mean residence time, distribution curve, variance and skewness. In order to give a better compatibility of the measurements at different process parameters, these moment methods are converted into their dimensionless forms and described as follows [7]:

$$\text{Dimensionless time} : \theta = \frac{t_i}{\tau} \quad (1)$$

where

$$\tau = \frac{\sum_i t_i C_i \Delta t_i}{\sum_i C_i \Delta t_i}, \text{ mean residence time} \quad (2)$$

$C_i$  is the existing tracer concentration at time  $t_i$  and  $\Delta t_i$  is the interval between two measurements.

Distribution curve:

$$E(\Theta) = \tau E(t) = \tau \frac{C_i}{\sum_i C_i \Delta t_i} \quad (3)$$

Variance:

$$\sigma(\Theta)^2 = \frac{\sigma(t)^2}{\tau^2} = \frac{\sum_i (t_i - \tau)^2 E(t) \Delta t_i}{\tau^2} \quad (4)$$

Skewness:

$$s = \frac{s(t)^3}{\sigma(t)^3} = \frac{\sum_i (t_i - \tau)^3 E(t) \Delta t_i}{(\sum_i (t_i - \tau)^2 E(t) \Delta t_i)^{3/2}} \quad (5)$$

### 2.3. Tanks-in-series model

The tanks-in-series model is a simple, but effective and robust model to characterise the fluid mixing in a reactor. The model assumes that the reactor acts as a series of  $N$  equal-sized tanks [7]. The age exit distribution ( $E$ ) for  $N$  tanks-in-series under the pulse injection is determined as follows:

$$E(t) = \frac{C(t)}{\int_0^\infty C(t) dt} = \frac{t^{(N-1)}}{(N-1)! \tau_i^N} e^{-t/\tau_i} \quad (6)$$

where  $\tau_i$  is the mean residence time for the  $i$ -th tank.

The mean residence time is defined:

$$\tau = \frac{\int_0^\infty t C(t) dt}{\int_0^\infty C(t) dt} = N \tau_i \quad (7)$$

In dimensionless form:

$$\Theta = \frac{t}{\tau} = \frac{t}{N \tau_i} \quad (8)$$

$$E(\Theta) = \tau E(t) = \frac{N(N\Theta)^{N-1}}{(N-1)!} e^{-N\Theta} \quad (9)$$

The number of tanks ( $N$ ) can be estimated as follows:

$$\sigma(\Theta)^2 = \frac{\sigma(t)^2}{\tau^2} = \int_0^\infty (\Theta - 1)^2 E(\Theta) d\Theta = \frac{1}{N} \quad (10)$$

$$N = \frac{1}{\sigma(\Theta)^2} \quad (11)$$

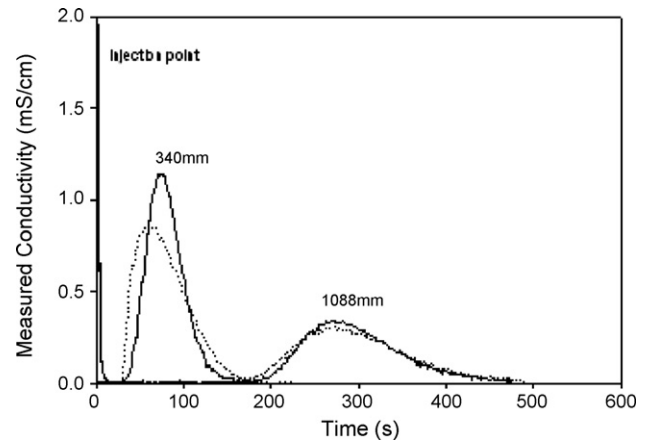


Fig. 2. RTD profiles from the central baffled reactor at different measuring points (dotted line: no oscillatory motion and solid line: oscillatory motion applied).

The number of tanks ( $N$ ) is determined by matching the model curve (Eq. (9)) with the experimental curve (Eq. (3)) as closely as possible in terms of shape, spread and height of the distribution. The method of least squares was used in this case. The aim is to minimise the sum of squared differences between the measurements (Eq. (3)) and the calculated values (Eq. (9)) for a particular value of  $N$ . Therefore, the initial estimated value of  $N$  was calculated using Eq. (11) based on the experimental variance (Eq. (4)). The value of  $N$  can be changed until the best correspondence is achieved.

## 3. Results and discussion

The RTD plays an important role in reactor characterisation as it relates to the mixing conditions inside a reactor. RTD curves vary from very narrow, symmetric distributions (plug flow) when there is substantial radial mixing, to, at the other extreme RTDs approximating those of continuous stirred tanks, when radial mixing is poor. A narrow RTD is normally preferred for continuous tubular reactor operation and therefore a lower variance is preferred.

### 3.1. RTD profiles

Plots of tracer concentration at various points along the reactor length versus time, with and without oscillatory motion, of a central baffled mesoreactor are presented in Fig. 2. In the presence of an oscillatory flow, a sharp and symmetrical RTD was observed at different measuring points responding to a near-pulse injection. However, the distribution of the tracer slightly broadened and had a longer tail on the right when the superimposed oscillatory flow was

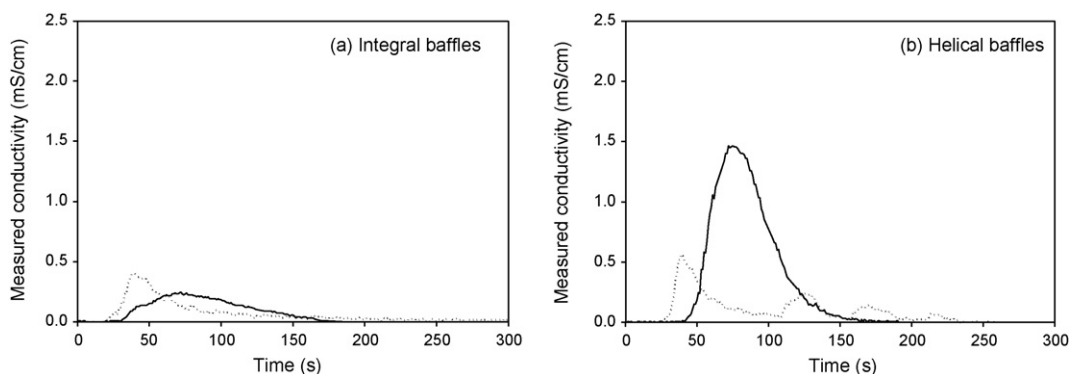


Fig. 3. RTD profiles at a measuring point of 340 mm at injection point (dotted line: no oscillatory motion and solid line: oscillatory motion applied). (a) Integral baffles and (b) helical baffles.

absent. This broadening is due to convection dispersion in laminar flow. This effect can be clearly seen in all the baffle designs.

Other phenomena are apparent in the RTDs of the other baffle designs. The RTD curves for the integral baffles without oscillation are clearly strongly tailed (Fig. 3(a)). The RTD curves for the helical baffled design is even more markedly divergent from plug flow in that its long tail has multiple peaks (Fig. 3(b)). These phenomena are due to the existence of stagnant zones and/or division of fluid flow. For both cases, the fluid in the central regions moves much faster than that in the wall region. Moreover, the interactions between the flow and sharp-edge of periodic helical baffles alter the flow direction, causing internal circulation. A further investigation of flow patterns in the helical baffled reactor needs to be carried out.

Generally speaking, the three continuous mesoscale oscillatory baffled reactors provided a well-defined and symmetrical RTD when an oscillatory flow was applied, which is the same behaviour as a conventional sharp-edged baffle reactor.

### 3.2. Effect of oscillation conditions on RTD performance

RTD profiles obtained from the three baffle designs are shown in Fig. 4 at a given net flow of 1.7 ml/min ( $Re_n = 7.2$ ) and amplitude of 1 mm ( $St = 0.4$ ). The variance of the RTD exhibits a maximum with respect to oscillation frequency. For example, at a frequency of 1 Hz, the RTD in the helical baffled reactor was close to that of a single stirred tank reactor, corresponding to a variance of 0.213. This value decreased significantly when increasing the frequency. A further increase in the frequency above 4 Hz caused the RTD to slightly broaden as shown in Fig. 4(b1), leading to a rise in variance as listed in Table 1. The narrowest RTD curve was observed at  $f = 4$  Hz for the central baffles, giving the smallest variance of 0.052.

The effect of oscillation amplitude on the RTD was also studied. In the case of the helical baffles, at a given frequency of 3 Hz, RTD profiles were close to Gaussian for all tested amplitude values from 1 to 4 mm (Fig. 4(b2)). Increasing the oscillatory amplitude

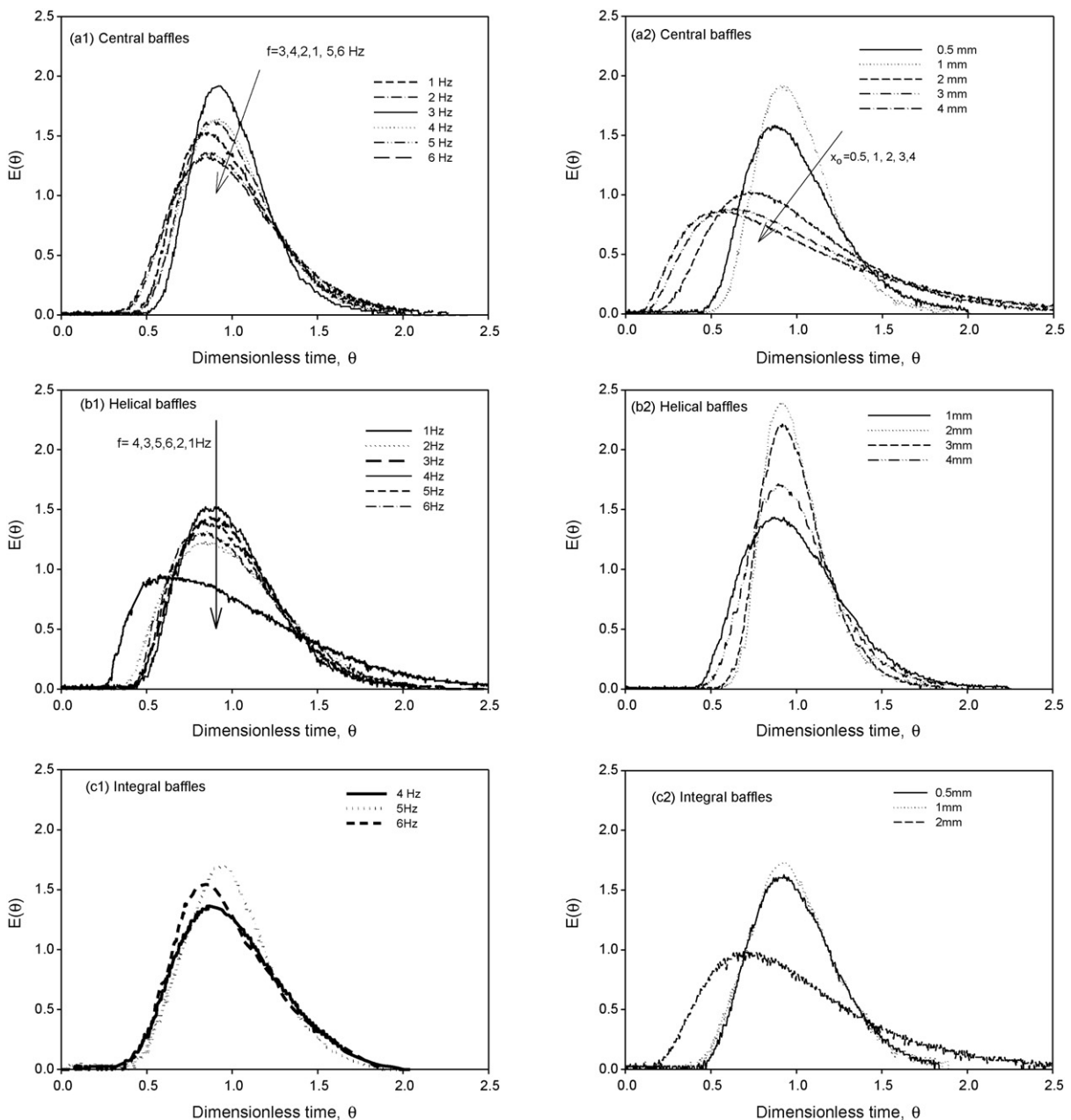


Fig. 4. Effect of (a1, b1, c1) frequency and (a2, b2, c2) amplitude on RTD profiles.

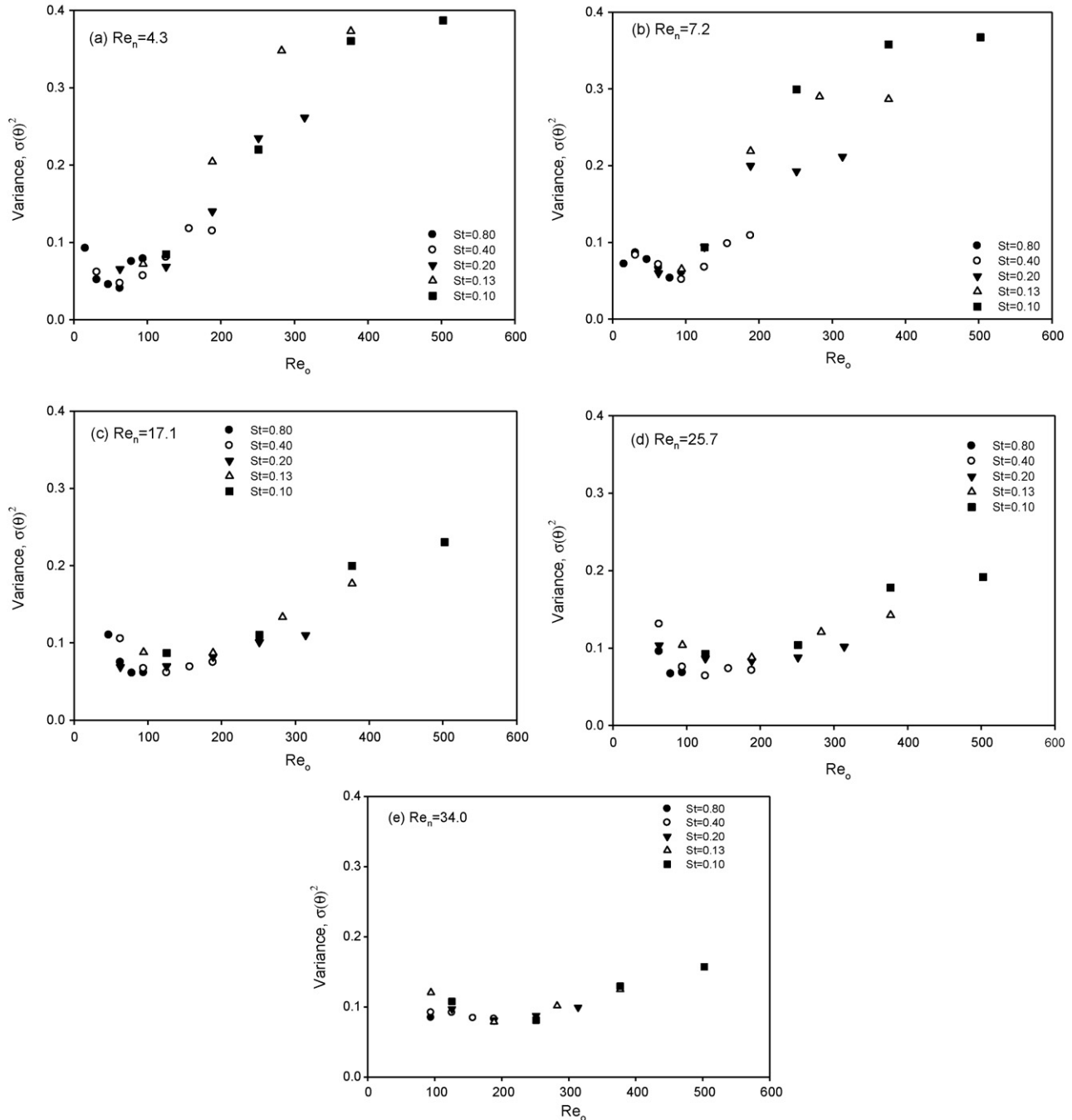
**Table 1**  
Effect of frequency on characteristics of RTD: variance (flow rate  $Q = 1.7$  ml/min and amplitude  $x_0 = 1$  mm).

Frequency (Hz)	Helical baffles	Central baffles	Integral baffles
1	0.213	0.083	0.1438
2	0.107	0.071	0.0865
3	0.088	0.052	0.0726
4	0.080	0.068	0.082
5	0.093	0.098	0.073
6	0.099	0.109	0.101

**Table 2**  
Effect of amplitude on characteristics of RTD: variance (flow rate  $Q = 1.7$  ml/min and frequency  $f = 3$  Hz).

Amplitude (mm)	Strouhal number	Helical baffles	Central baffles	Integral baffles <sup>a</sup>
0.5	0.80	0.103	0.078	0.068
1	0.40	0.088	0.052	0.073
2	0.20	0.034	0.200	0.132
3	0.13	0.041	0.290	0.172
4	0.10	0.065	0.358	0.223

<sup>a</sup> Frequency: 5 Hz.



**Fig. 5.** Variances obtained from RTD profiles at various oscillation conditions with net flow Reynolds number: (a)  $Re_n = 4.3$ ; (b)  $Re_n = 7.2$ ; (c)  $Re_n = 17.1$ ; (d)  $Re_n = 25.7$ ; and (e)  $Re_n = 34.0$ .



narrowed the RTD. As shown in Fig. 4(b2), the distribution was narrower for amplitudes above 2 mm than for 1 mm. This can also be observed in Table 2, where the variance decreased from 0.088 at 1 mm amplitude to 0.034 at 2 mm amplitude. For the case of the central and integral baffles, the Gaussian form was only maintained at amplitudes of up to 1 mm (Fig. 4(a2) and (c2)). At higher amplitudes, the RTD profiles approximated a completely mixed state, indicating that axial mixing becomes dominant at these amplitudes. This is because oscillation amplitude directly relates to the length of eddies propagated inside each baffled cavity. Previous studies [1,15] found that an increase in oscillation amplitude resulted in an increase in the size of eddies generated. At higher amplitudes the vortices travel too far, such that they interact with

adjacent baffles, thereby rendering the flow less like discrete tanks-in-series. This can be prevented in practice by observance of design rules based on maintaining the Strouhal number above certain values (see Table 2 below).

### 3.3. Interdependence of net flow and oscillatory flow on RTD performance

The mean residence time ( $\tau$ ), which was calculated using Eq. (2), was not affected by oscillation conditions. The residence times used were 340, 90 and 50 s, resulting in  $Re_n$  of 4.3, 17.1 and 34.0, respectively. Compared to the corresponding hydraulic residence time (which is calculated by dividing the reactor volume to the

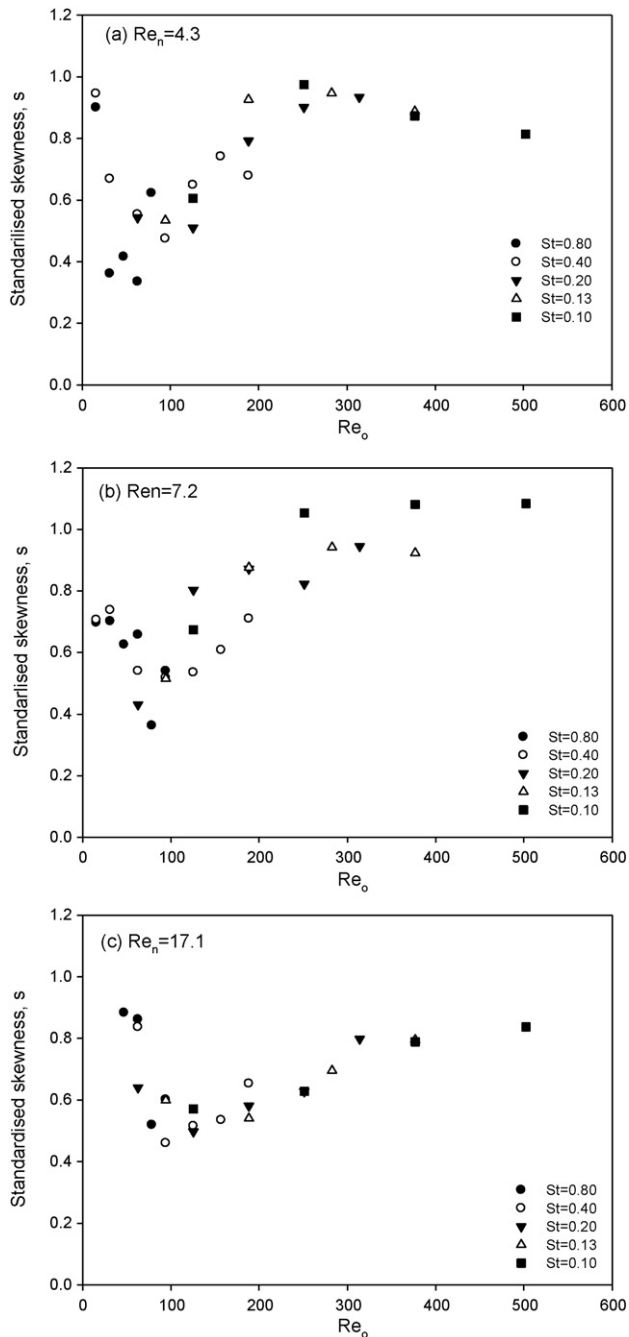


Fig. 6. Skewness obtained from RTD profiles at various oscillation conditions with net flow Reynolds number: (a)  $Re_n = 4.3$ ; (b)  $Re_n = 7.2$ ; and (c)  $Re_n = 17.1$ .

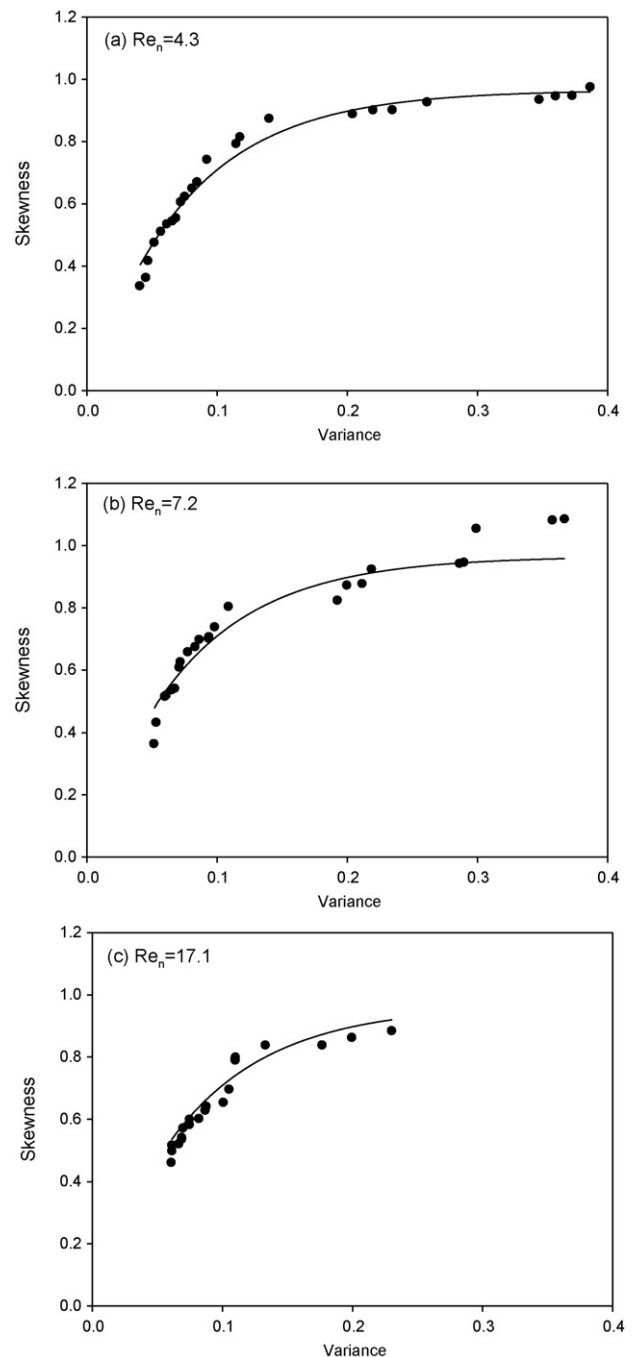


Fig. 7. Correlation skewness versus variance according to Eq. (12).

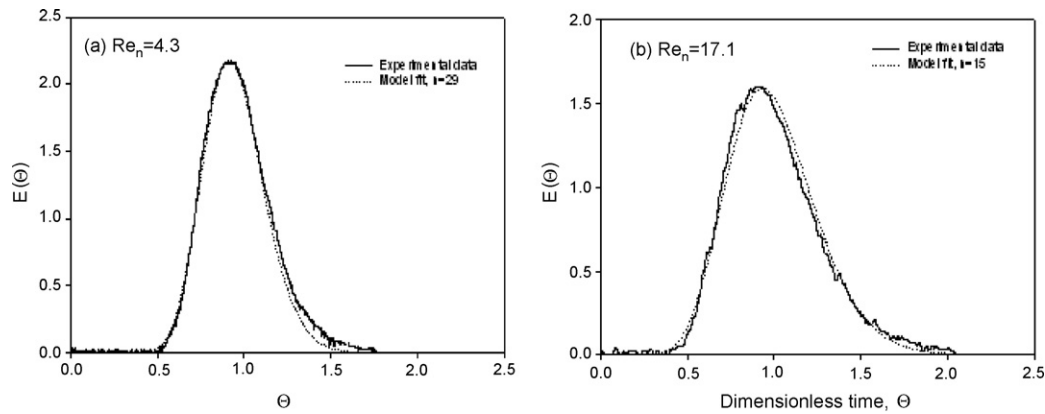


Fig. 8. RTD profiles obtained from experiments (solid line) and number of tanks-in-series model at net flow Reynolds numbers of (a)  $Re_n = 4.3$  and (b)  $Re_n = 17.1$ .

flow rate:  $\tau = V/Q$ , the mean residence time was higher, 8.5% at net flow Reynolds numbers of 4.3 and 7.2, and 14–22% at higher net flow Reynolds numbers. The prolonged mean residence time was similar to the findings of others [2,14]. Reis et al. [14] found that the difference between the average mean residence time and the hydraulic time was 32.5% at a flow rate of 1.94 ml/min. Harvey et al. [2] observed that the residence time of bubbles increased significantly when the fluid was oscillated at optimal oscillation conditions.

The dependency of RTD on oscillation conditions at different net flow rates is shown in Fig. 5. At  $Re_n < 10$ , oscillation conditions had a strong influence on the mixing inside the reactor. The variances decreased to minima at a critical value of  $Re_o \sim 50$  (Fig. 5(a)) and then increased sharply with a further increase in  $Re_o$ . Skewness is the parameter to measure asymmetric of the RTD whilst the variance measures the spread of the distribution. Skewness can be positive (longer tail to the right of the mean), negative (longer tail to the left of the mean) or zero (symmetric distribution). As shown in Fig. 6, the lowest skewnesses are also observed at these critical  $Re_o$ . At  $17 \leq Re_n < 30$ , there was little effect of oscillation conditions on the RTD behaviour. The deviation of variances was small compared to the cases of low net flows ( $Re_n = 4.3$  and 7.2). For example, the variance increased slightly from a minimum value of around 0.06–0.2 for the case of  $Re_n = 17.1$  whilst it rose rapidly from 0.05 to 0.4 for the case of  $Re_n = 4.3$  with increasing  $Re_o$ . At  $Re_n$  above 30, the variances remained at approximately 0.1 regardless of increases in the value of  $Re_o$  (Fig. 5(e)). The correlation of variance ( $\sigma(\theta)^2$ ) versus skewness ( $s$ ) is also established as expressed:

$$s = \frac{0.9311 \times [1 - \exp[-12.3944 \times (\sigma(\theta)^2)]]}{Re_n^{-0.0442}} \quad (12)$$

Fig. 7 shows a good agreement between experimental data and fitted correlation data. At a fixed net flow, skewness increased exponentially with increasing variances or equivalently to a reduction of number of tanks as the reactor behaved similarly to a single stirred tank.

Generally, the RTD behaviour was a function of the oscillation conditions and net flow, which is similar to the findings of Stonestreet and Van Der Veecken [20] for conventional OBRs. At given oscillation conditions ( $Re_o$ ), increasing  $Re_n$  led to wider, less symmetric distributions. However, the near-Gaussian form of the RTD can be maintained by controlling the oscillation amplitude and frequency, increasing  $Re_o$ . At  $Re_n$  above 25, the oscillation conditions had little effect on the RTD curves, in terms of the spread of the RTD.

#### 4. Dependence of the fluid mixing on the velocity ratio of oscillatory flow Reynolds number ( $Re_o$ ) to net flow Reynolds number ( $Re_n$ )

The interaction between the sharp-edged baffles and oscillating fluid provides radial mixing by repeatedly forming toroidal vortices. These radial velocities give uniform mixing in each inter-baffle zone and cumulatively along the length of the column. Therefore, each baffle cavity is considered to act as a small stirred tank reactor. This results in a narrow RTD due to having many “tanks” in series. For instance, the single tube in this work contains 39 baffle cavities.

Fig. 8 shows the number of tanks-in-series obtained from the central baffled reactor operating at different oscillation conditions and net Reynolds numbers, compared to curves that would result from various series of perfect stirred tanks-in-series. Broadly, the curves match well with the experimental ones in terms of height, shape and spread of the distribution. This indicates that the tanks-in-series model is valid for this reactor.

To take into account the interdependence of  $Re_o$  and  $Re_n$  on the mixing conditions inside the mesoscale OBRs, the “velocity ratio”,  $\psi$  (the ratio of oscillatory Reynolds number to net Reynolds number) was investigated. Fig. 9 shows the number of tanks versus velocity ratio for five different cases of net flows ( $Re_n$ s) over a wide range of frequencies and amplitudes. It was observed that the trend of the dependency of the number of tanks on the velocity ratio was similar

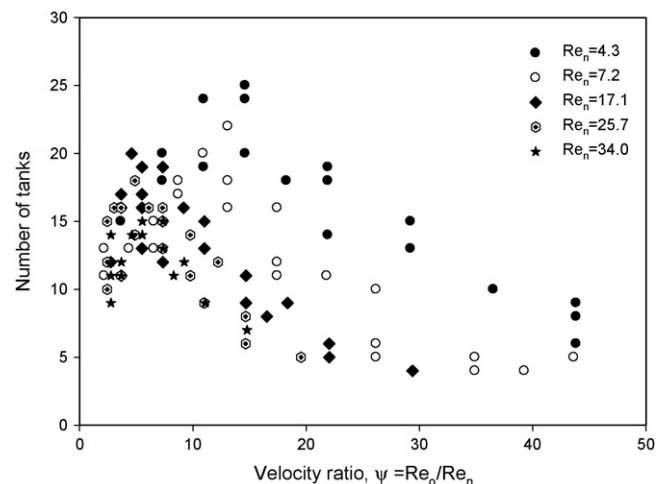


Fig. 9. Dependence of RTD performances on velocity ratio for the central baffled reactor.

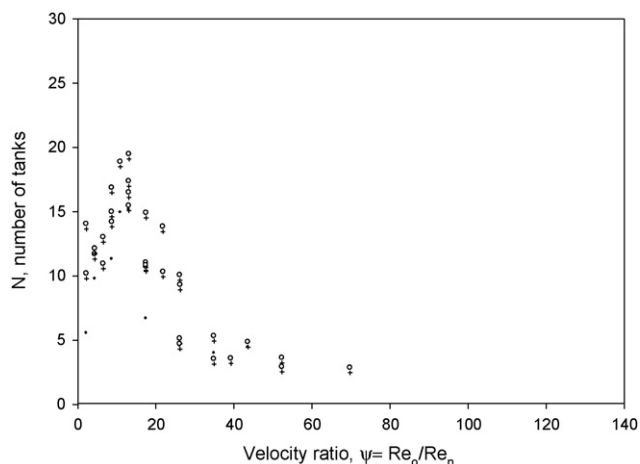


Fig. 10. Dependence of RTD performances on velocity ratio for the integral baffled reactor.

to that in conventional scale OBRs [20]. In the central baffle design, the number of tanks-in-series increased with velocity ratio to a maximum at 8, and then decreased monotonically. An acceptable level of plug flow in practice is 10 tanks-in-series [3]. In order to obtain a value of  $N$  above 10, the velocity ratio must be in the range 4–8.

The number of tanks-in-series in the integral baffled reactor is also presented in Fig. 10. The number of tanks reached its maximum value at a velocity ratio of 5–10 and oscillation amplitude of 0.5–1 mm and subsequently decreased with an increase in velocity.

## 5. Conclusions

Three designs of oscillatory baffled meso-reactors were constructed and their RTD performance characterised at low net flow rates ( $Re_n$  below 50). The oscillation conditions were shown to have a strong influence on the RTD at  $Re_n < 10$  and little effect on the RTD curves at  $Re_n > 25$ . The critical value of  $Re_o$  (where the minimum variance and skewness was achieved) was shown to be a function of  $Re_n$ . Increasing  $Re_n$  increased the critical  $Re_o$  was 30–50 at  $Re_n = 4.3$ , but was 70–100 at  $Re_n = 7.2$ . Nonetheless, the minimum variance and skewness were obtained at a similar velocity ratio of oscillatory Reynolds number to net flow Reynolds number. At a given  $Re_n$ , the greatest level of plug flow is achieved at a velocity ratio of 4–8 for the central baffle design and 5–10 for the integral baffle design.

The helical baffled reactor performs at near plug flow at higher oscillation amplitudes, i.e. 2–4 mm compared to the other baffled designs, which was at 0.5–1 mm. The differences in behaviour of the helical baffled design necessitate further investigation, as many of the characteristics, particularly the wider operating window, are desirable.

Overall, it has been demonstrated that all three baffle designs considered can be used to produce plug flow at flow rates varying 1.0–8.0 ml/min. Choosing between these three designs will depend upon the application, and could be based upon e.g. the ease of degassing, fabrication or cleaning, but all are easily fabricated and practical at this scale, whereas the manufacture of conventional orifice plate baffles on supporting rods was found to be impractical at this scale.

The results from this work can be used to inform the design and operation of mesoscale oscillatory baffled reactors. This reac-

tor platform can be used to screen processes, perhaps for very small production, but unlike most other designs of laboratory-scale continuous reactor (for the central baffled and integral baffled designs at least). It has been shown that conventional OBRs can be predictably scaled up directly from laboratory scale to industrial scale [6,16]. The findings in this study indicated that the central and integral baffled designs behaved similarly to conventional OBRs. This implies that scale-up to industrial scale is feasible here. However, further investigation should be carried out. The helical baffle design is not yet proven at other scales, but this work is planned.

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