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Experimental investigation of the effect of scale-up on mixing efficiency in oscillatory flow baffled reactors (OFBR) using principal component based image analysis as a novel noninvasive residence time distribution measurement approach

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HIGHLIGHTS

- Oscillation amplitude positively correlates with system dispersion.
- Oscillation frequency is loosely correlated with system dispersion.
- Piston driven flow leads to suboptimal operation while pump driven flow is desired.
- Optimums for oscillatory parameters exist and depend on system scale.
- Principal component based image analysis offers process flexibility.

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ABSTRACT

Oscillatory flow strategies through baffled tubular reactors provide an efficient approach in improving process kinetics through enhanced micromixing and heat transfer. Known to have high surface area to volume ratios, oscillatory flow baffled reactors (OFBR) generate turbulence by superimposing piston driven oscillatory flow onto the net flow generated by a pump. By tuning the oscillating parameters (amplitude and frequency), one can tailor the residence time distribution of the system for a variety of multiphase applications. Using a microscope camera, principal component image analysis, and pulse tracer injections, a novel noncontact approach has been developed to experimentally estimate dispersion coefficients in two geometrically different systems (DN6 and DN15, Alconbury Weston Ltd.). The paper also introduces for the first time a novel scaled-down version of the commercially available DN15 OFBR, the DN6 (about 10 times smaller scale), and provides a comprehensive investigation of the effect of oscillation parameters on the residence time distributions (RTD) in both systems. The oscillation amplitude was found to have a significant positive correlation with the dispersion coefficient with 1 mm providing the least amount of dispersion in either system. Oscillation frequency had a less significant impact on the dispersion coefficient, but optimal operation was found to occur at 1.5 Hz for the DN6 and 1.0 Hz for the DN15. Until now, OFBR literature has not distinguished between piston and pump driven flow. Pump driven flow was found to be ideal for both systems as it minimizes the measured dispersion coefficient. However, piston driven turbulence is essential for avoiding particle settling in multi-phase (solid-liquid) systems and should be considered in applications like crystallization.

1. Introduction

Increasingly popular for both synthesis and purification applications, continuous oscillatory flow strategies improve process kinetics through enhanced micromixing and heat transfer characteristics [1]. Specifically useful for dual phase systems (liquid-liquid/solid-liquid), oscillatory flow baffled reactors (OFBR) offer process flexibility with adaptable configurations. OFBRs are often divided into several zones which allow for the implementation of temperature profiles and the spatial distribution of reagents across multiple injection points [1].

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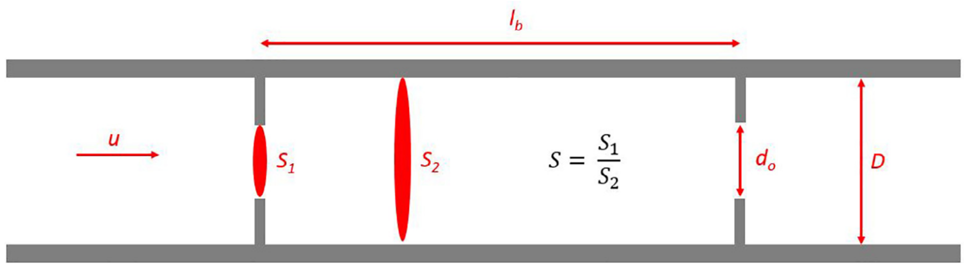

Fig. 1. Illustration of OFBC geometry. Adapted from McDonough [7].

Table 1
Optimum operating parameters of OFBRs in literature.

System	DN15	Custom	Custom	Amicon-Wright
Length (m)	~5	5	20.5	0.67
Inner Diameter (mm)	15	25	40	23
Vertical (V) or Horizontal (H)	V	V	V	H
Re_o	94–3000	435	2008	Not reported
Optimal Frequency (Hz)	Both high (5) and low (1)	Not reported	2	No effect
Optimal Amplitude (mm)	1	Not reported	4	1
Conclusions	Ψ is not sufficient in characterizing a system	Scale up can be achieved using a multi-tube configuration	Increasing x_0 led to increasing dispersion	Frequency had an insignificant effect on dispersion measurements
Author	Kacker [4]	Ni [15]	Pereira [6]	Dickens [5]

These added degrees of freedom provide numerous design advantages when compared to their continuous stirred tank counterparts.

Compared to traditional plug flow reactors (PFRs), OFBRs generate turbulence by superimposing an oscillatory flow onto the net flow through the use of a piston. By imposing this oscillatory turbulence, OFBRs do not need to operate at high throughput flow rates like PFRs, meaning smaller tube lengths and holdup volumes. However, several authors have indicated that these oscillations have a significant impact on the RTD of the system [2–6], but do not consider system geometry or scale up. Herein, two commercial systems of different geometry and scale will be evaluated under a variety of operating conditions.

Conventional oscillatory baffled reactors vary in geometry and have diameters of 15 mm or greater [7], while the newer (more recent) mesoscale reactors have diameters in the range of 4–5 mm. Regardless of scale, the commercial application of oscillatory systems are generally limited to purely liquid systems, as plug flow operation in a two phase (solid/liquid) system is still mainly a topic of interest amongst academics, especially for shear-sensitive applications [8].

The flow characteristics in oscillatory systems are largely governed by both operating and geometric parameters [9]. As described in Fig. 1, *S* controls the size and shape of eddies formed, while adequate distance between baffles (l_b) ensures fully developed vortices and the minimization of mixing dead zones [10,11]. Baffle type also plays a major role and should be chosen appropriately for a given application. For example, a major concern in designing crystallization systems is minimizing shear stress, as it can lead to crystal breakage and broad size distributions [12]. Integral baffles (like those in the DN6 and DN15) provide a low shear environment [13,14] by smoothly constricting the inner diameter of the tube periodically, making them ideal for these applications (See Figs. 2–6).

Four dimensionless parameters define the fluid flow patterns in an OBC, that is, the oscillatory Reynolds number (Re_o), the net flow Reynolds number (Re_n), the Strouhal number (St), and ψ, the ratio of oscillatory and net flows.

$$Re_o = \frac{2\pi f x_0 \rho D}{\mu} \tag{1}$$

$$Re_n = \frac{\rho u D}{\mu} \tag{2}$$

$$St = \frac{D}{4\pi x_0} \tag{3}$$

$$\psi = \frac{Re_o}{Re_n} \tag{4}$$

Note: x_0 is the piston amplitude, ρ is the solution density, D is the diameter of the tube segment, u is the mean superficial velocity and μ is the solution viscosity. The St measures effective eddy propagation by simply taking the ratio of the column diameter to piston stroke length [6]. The Re_o represents the turbulence generated by the oscillating piston. Similar to traditional fluid dynamics, the Re_n is a ratio of inertial to viscous forces. The ratio of oscillatory and net flow Re result in the overall mixing parameter ψ, a commonly used turbulence parameter.

2. Residence time distributions in oscillatory baffled systems

A major advantage in oscillatory tubular systems is the ability to operate near plug flow for narrow residence time distributions [16]. As seen in Table 1, several authors have worked to develop the operating framework for continuous oscillatory baffled systems. Each had a geometrically different system, but the trends for optimal operation are similar. Kacker's work used the commercially available DN15 manufactured by Alconbury Weston Ltd. He found that Ψ is not a sufficient parameter in characterizing the fluid dynamics of their system and that optimal operation occurred at both high and low frequencies as long as the amplitude remained small (1 mm). Similarly, Dickens reported the same optimal amplitude of oscillation and that frequency had little to no effect on dispersion measurements. Moreover, both Kacker's and Dickens' results agree with those conducted by Pereira, who showed that increasing the amplitude of oscillation resulted in increased dispersion and a larger mean residence time. Ni used flow visualization studies to evaluate the benefits of both baffles and oscillations in achieving plug flow behavior at minimal flow rates (Re_N). Plug flow operation is vital for crystallization, as narrow CSDs are directly proportional to narrow RTDs. Moreover, the majority of RTDs in OFBR literature are conducted for single phase liquid systems, which do not accurately represent solid-liquid systems. Kacker, however, performed both liquid and solid-liquid RTD studies and showed that plug flow operation is achievable under a variety of conditions Table 2.