EPSRC
Centre for Innovative Manufacturing
in Continuous Manufacturing and Crystallisation

A design of experiment (DoE) approach to optimize the inner geometry of baffled meso-scale tubes for continuous crystallization

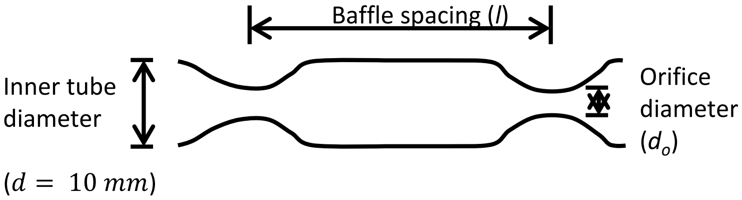
Louisa Ejim (Loughborough University)

Supervisor: Dr. Nuno Reis

2nd Supervisor: Prof. Chris D. Rielly

EPSRC
Engineering and Physical Sciences
Research Council

Aim & Objectives

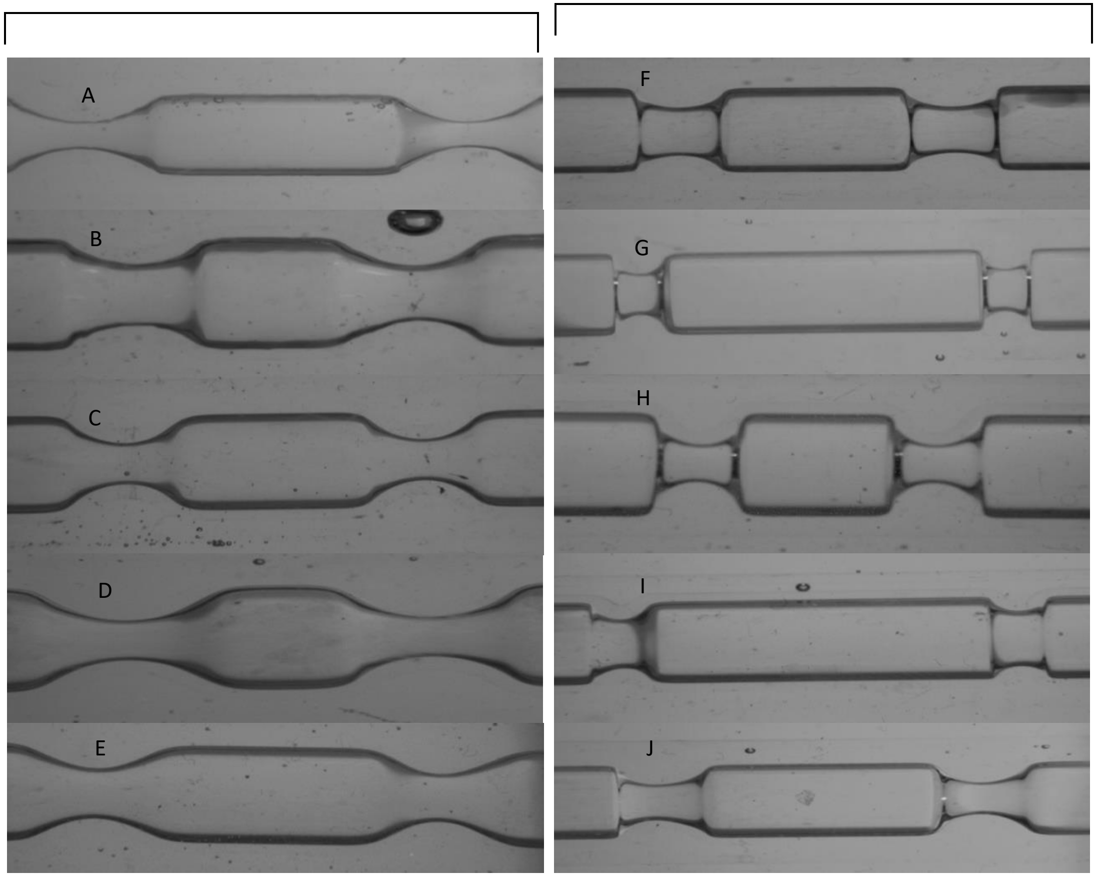


- To determine the optimal geometry
- By characterising the tubes using solid-liquid residence time distribution (RTD)
- DoE approach to investigate the effect of baffle spacing (l), orifice diameter (d_o) and baffle type on RTD
- PVC particles are used because of their similarities in flow properties to crystals.

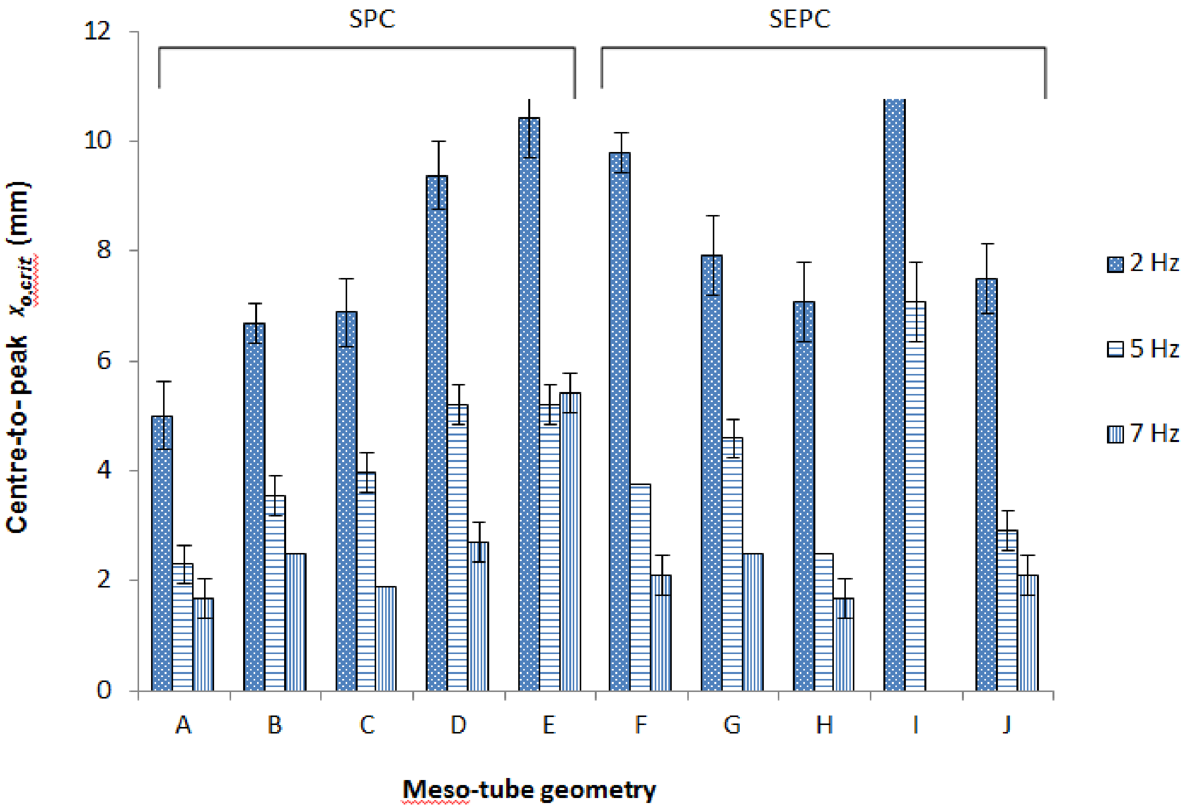
Glass tubes;

Smooth edged baffle type (SPC)

Sharp edged baffle type (SEPC)



Batch Suspension Studies (critical amplitude: centre-to-peak)

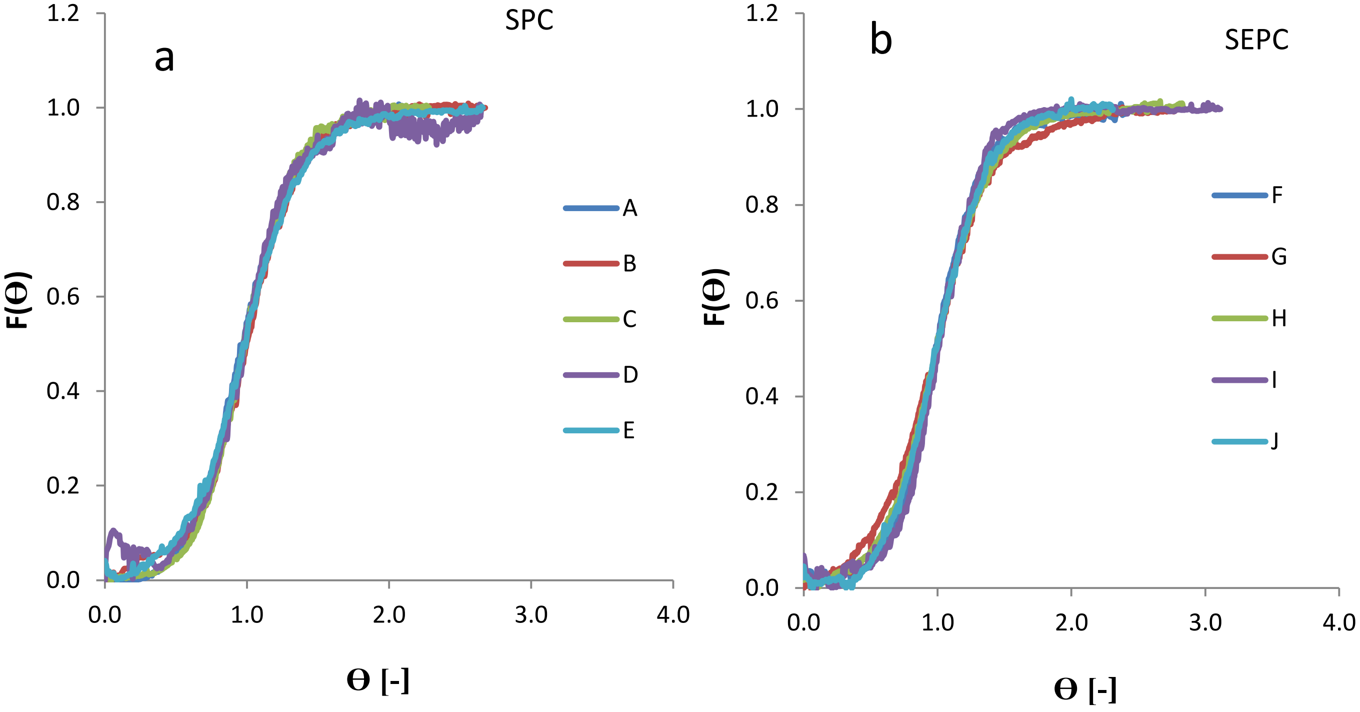


Conclusions:

- Increased oscillation frequency provides a lower critical amplitude required to suspend particles
- Sharp edged-baffles of the same dimensions proved to require higher critical amplitude
- **Tube *A*** ($l = 30$ mm, d_o= 3.5 mm) proved to require the lowest amplitude for suspension.

Continuous Liquid-solid RTD

$$W(t) = \frac{C_{out}(t)}{C_0}\ ,\ F(t) = 1 - W\ (t)\ ,\ \ \theta = \frac{t}{\bar{t}}$$



Conclusions:

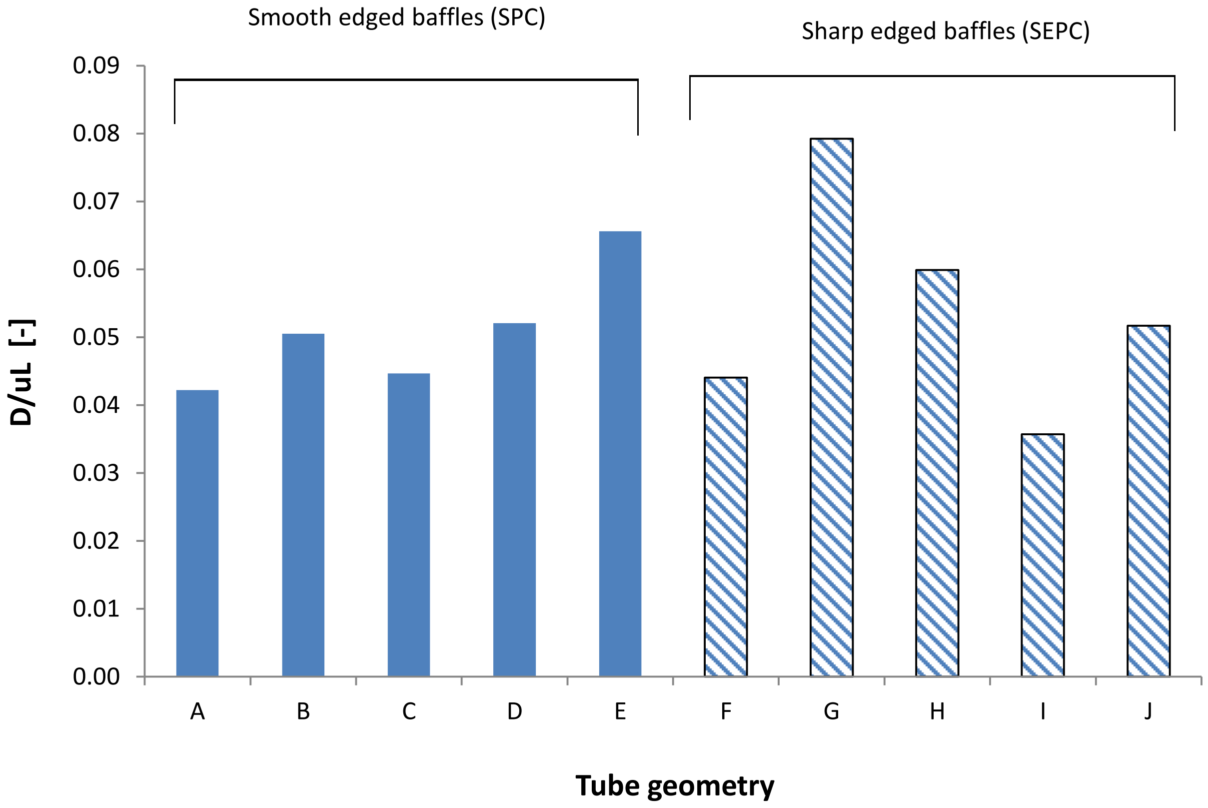
Smooth-edged baffle type:

- Gives lower energy input requirement (e.g critical amplitude)
- Narrower RTD (close to plug flow behaviour)
- Uniform mixing

Sharp- edged baffle type:

- Chaotic mixing
- Broader RTD
- Bubble retention/trapping.

Plug flow with axial dispersion (D/uL)



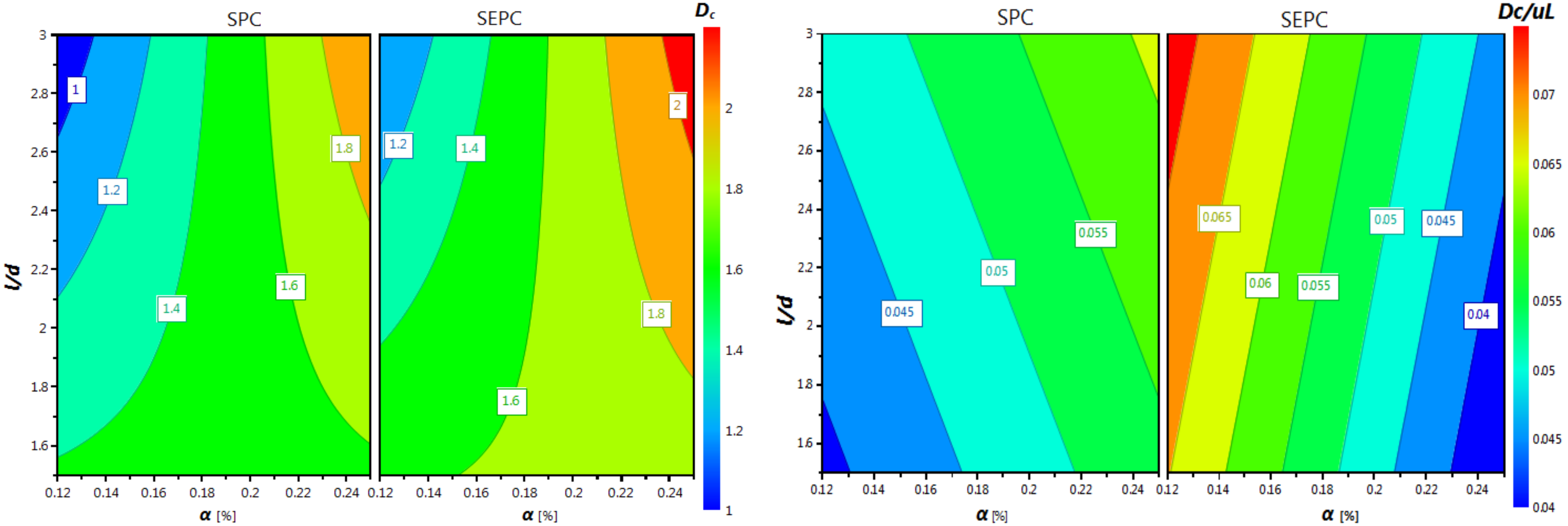
- ➢ Levenspiel Open- Open boundary condition:

$$E(\theta) = \frac{1}{\sqrt{4\pi(D_c/uL)}} exp\left[-\frac{(1-\theta)^2}{4\theta(D_c/uL)}\right]$$

Fitting parameters:

- Superficial velocity (m/s)
- Axial dispersion coefficient (m^2/s).

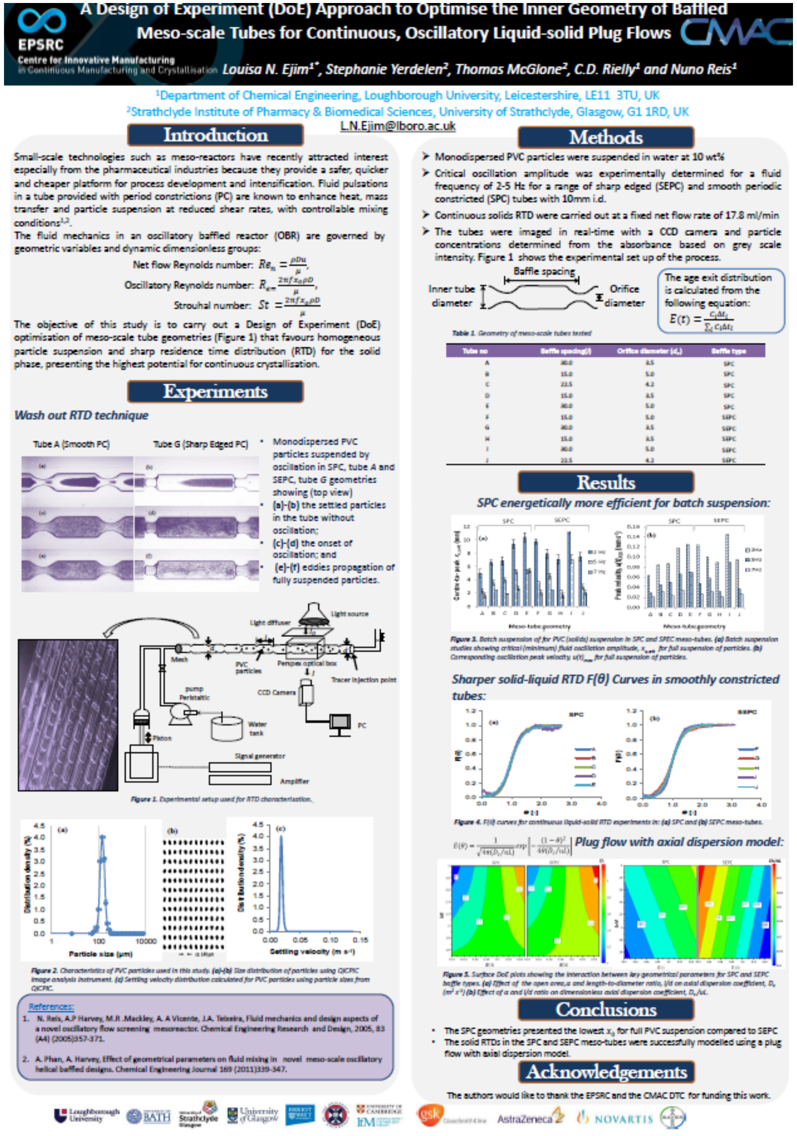
Plug flow with axial dispersion DoE plots



Conclusion

- The open baffle area, α identified as the dominant design parameter in controlling solids backmixing and batch suspension of particles, with small values of $\alpha = 0.12$ resulting in minimised axial dispersion
- Strong eddy vortices generated at lower values of α, that presumably led to trapping of particles, an effect not previously observed in OFRs
- Tube ***A*** (*l*/*d* = 3.0) showed clear flow characteristic advantages over the other meso-tubes studied.

Thank You!



A Design of Experiment (DoE) Approach to Optimise the Inner Geometry of Baffled Meso-scale Tubes for Continuous, Oscillatory Liquid-solid Plug Flows

Louisa N. Ejim[1], Stephanie Yerdelen[2], Thomas McGlone[2], C.D. Rielly[1] and Nuno Reis[1]*

[1]Department of Chemical Engineering, Loughborough University, Leicestershire, LE11 3TU, UK
[2]Strathclyde Institute of Pharmacy & Biomedical Sciences, University of Strathclyde, Glasgow, G1 1RD, UK
L.N.Ejim@lboro.ac.uk

Introduction

Small-scale technologies such as meso-reactors have recently attracted interest especially from the pharmaceutical industries because they provide a safer, quicker and cheaper platform for process development and intensification. Fluid pulsations in a tube provided with period constrictions (PC) are known to enhance heat, mass transfer and particle suspension at reduced shear rates, with controllable mixing conditions[1,2].

The fluid mechanics in an oscillatory baffled reactor (OBR) are governed by geometric variables and dynamic dimensionless groups:

Net flow Reynolds number: $Re_n = \frac{\rho D u}{\mu}$,

Oscillatory Reynolds number: $R_{eo} = \frac{2\pi f x_o \rho D}{\mu}$,

Strouhal number: $St = \frac{2\pi f x_o \rho D}{\mu}$

The objective of this study is to carry out a Design of Experiment (DoE) optimisation of meso-scale tube geometries (Figure 1) that favours homogeneous particle suspension and sharp residence time distribution (RTD) for the solid phase, presenting the highest potential for continuous crystallisation.

Experiments

Wash out RTD technique

- Monodispersed PVC particles suspended by oscillation in SPC, tube *A* and SEPC, tube *G* geometries showing (top view)
- (a)-(b) the settled particles in the tube without oscillation;
- (c)-(d) the onset of oscillation; and
- (e)-(f) eddies propagation of fully suspended particles.

Figure 1. Experimental setup used for RTD characterisation.

Figure 2. Characteristics of PVC particles used in this study. (a)-(b) Size distribution of particles using QICPIC image analysis instrument. (c) Settling velocity distribution calculated for PVC particles using particle sizes from QICPIC.

References:

1. N. Reis, A.P Harvey, M.R .Mackley, A. A Vicente, J.A. Teixeira, Fluid mechanics and design aspects of a novel oscillatory flow screening mesoreactor. Chemical Engineering Research and Design, 2005, 83 (A4) (2005)357-371.
2. A. Phan, A. Harvey, Effect of geometrical parameters on fluid mixing in novel meso-scale oscillatory helical baffled designs. Chemical Engineering Journal 169 (2011)339-347.

Methods

- Monodispersed PVC particles were suspended in water at 10 wt%
- Critical oscillation amplitude was experimentally determined for a fluid frequency of 2-5 Hz for a range of sharp edged (SEPC) and smooth periodic constricted (SPC) tubes with 10mm i.d.
- Continuous solids RTD were carried out at a fixed net flow rate of 17.8 ml/min
- The tubes were imaged in real-time with a CCD camera and particle concentrations determined from the absorbance based on grey scale intensity. Figure 1 shows the experimental set up of the process.

The age exit distribution is calculated from the following equation:

$$E(t) = \frac{C_i \Delta t_i}{\sum_i C_i \Delta t_i}$$

Table 1. Geometry of meso-scale tubes tested

Tube no	Baffle spacing(*l*)	Orifice diameter (d_o)	Baffle type
A	30.0	3.5	SPC
B	15.0	5.0	SPC
C	22.5	4.2	SPC
D	15.0	3.5	SPC
E	30.0	5.0	SPC
F	15.0	5.0	SEPC
G	30.0	3.5	SEPC
H	15.0	3.5	SEPC
I	30.0	5.0	SEPC
J	22.5	4.2	SEPC

Results

SPC energetically more efficient for batch suspension:

Figure 3. Batch suspension of for PVC (solids) suspension in SPC and SPEC meso-tubes. (a) Batch suspension studies showing critical (minimum) fluid oscillation amplitude, $x_{o,crit}$ for full suspension of particles. (b) Corresponding oscillation peak velocity, $u(t)_{max}$ for full suspension of particles.

Sharper solid-liquid RTD F(θ) Curves in smoothly constricted tubes:

Figure 4. F(θ) curves for continuous liquid-solid RTD experiments in: (a) SPC and (b) SEPC meso-tubes.

$$E(\theta) = \frac{1}{\sqrt{4\pi(D_L/uL)}} exp\left[-\frac{(1-\theta)^2}{4\theta(D_L/uL)}\right]$$

Plug flow with axial dispersion model:

Figure 5. Surface DoE plots showing the interaction between key geometrical parameters for SPC and SEPC baffle types. (a) Effect of the open area, α and length-to-diameter ratio, l/d on axial dispersion coefficient, D_L ($m^2 s^{-1}$) (b) Effect of α and l/d ratio on dimensionless axial dispersion coefficient, D_L/uL.

Conclusions

- The SPC geometries presented the lowest x_o for full PVC suspension compared to SEPC
- The solid RTDs in the SPC and SEPC meso-tubes were successfully modelled using a plug flow with axial dispersion model.

Acknowledgements

The authors would like to thank the EPSRC and the CMAC DTC for funding this work.