Newcastle University ePrints

Mohammadi S, Boodhoo KVK. [Online conductivity measurement of residence time distribution of thin film flow in the spinning disc reactor.](#)

Chemical Engineering Journal 2012, 207-208, 885-894.

Copyright:

NOTICE: this is the author's version of a work that was accepted for publication in *Chemical Engineering Journal*. Changes resulting from the publishing process, such as peer review, editing, corrections, structural formatting, and other quality control mechanisms may not be reflected in this document. Changes may have been made to this work since it was submitted for publication. A definitive version was subsequently published in *Chemical Engineering Journal*, Volumes 207–208, 1 October 2012.
DOI: 10.1016/j.cej.2012.07.120

Further information on publisher website: <http://www.elsevier.com/>

Date deposited: 10th July 2014

Version of article: Author accepted manuscript

This work is licensed under a [Creative Commons Attribution-NonCommercial 3.0 Unported License](#)

ePrints – Newcastle University ePrints
<http://eprint.ncl.ac.uk>

Online conductivity measurement of residence time distribution of thin film flow in the spinning disc reactor

Somaieh Mohammadi* and Kamelia V.K. Boodhoo

School of Chemical Engineering & Advanced Materials,

Newcastle University, Merz Court,

Newcastle Upon Tyne NE1 7RU, UK

Corresponding author: s.mohmmadi@ncl.ac.uk

Tel: +44 191 222 7621; Fax: + 44 191 222 5292

Abstract

Spinning disc reactors provide effective continuous flow mixing in highly sheared liquid films flowing under the action of high centrifugal forces generated as a result of the disc rotation. Although the flow is theoretically characterised to be laminar on the basis of the low Reynolds numbers, the film surface is normally covered with numerous ripples which can change the film hydrodynamics and velocity distribution and thereby affect its residence time distribution (RTD) on the rotating disc. The aim of this study is to determine the experimental conditions for which near plug flow behaviour prevails on the spinning disc. Our findings indicate that the higher the disc speed and liquid flow rate and the lower the viscosity of the processing liquid, the closer conditions within the liquid film on the disc approximate to plug flow behaviour. These effects are attributed to the greater degree of turbulence induced in the liquid film as a result of an increased intensity of surface waves, promoting transverse mixing across the film thickness and thus a more uniform velocity profile at any given radial position. The centrifugal force directing the liquid radially to the disc periphery may also play a part in reducing radial dispersion. The texture of the disc surface is also an influential parameter in achieving plug flow on the rotating disc. With flow on a grooved disc, the number of tanks-in-series increased quite significantly under identical operating conditions as those for a smooth disc, confirming that radial dispersion is lowered. One of the explanations for this effect is that the constantly changing topography enabled by the series of concentric grooves across the disc surface offers the opportunity for repeated film detachment and re-attachment which cause induced recirculation or vortices within the film, giving better transverse mixing and eliminating velocity gradients.

Keywords

Spinning Disc Reactor; Residence Time Distribution; Plug flow; Radial dispersion; Peclet number; Tanks in series, Conductivity

1. Introduction

Spinning disc reactors (SDRs) have received considerable attention as an intensified reactor technology which can produce highly sheared thin films with enhanced heat and mass transfer characteristics [1-6]. As a consequence the SDR has been shown to be suitable for exothermic reactions [7-9], and to be very effective as continuous-flow mixers which can offer important benefits over traditional batch processing equipment for intrinsically fast reactions [1]. In addition, the control of particle size distribution for fast crystallisations in the SDR has been reported [10]. Crystallization processes in the pharmaceutical industry are usually intended to achieve crystals with a controlled size, shape, purity, and polymorphic form [11].

Consistent product quality can only be achieved in plug flow reactors and plug flow conditions are beneficial for higher conversion rates and shorter residence times [12]. Fast processes, such as nucleation during crystallization, are typically highly dependent on mixing and imperfect crystallization can be caused by non-uniform and slow mixing. The effect of mixing on crystallisation is reflected on two scales: micro and macro-mixing. Micromixing relates to mixing at the molecular level. In fast chemical processes like precipitation, the effect of micromixing is significant. Macromixing, associated with residence time distributions, equalizes the concentrations of all the species within the reaction volume. Residence time is different for each solution, and for particles of different sizes [13]. In studies of crystallisation, determination of the RTD is routine, especially for non-Newtonian and viscous products [14]. RTD delivers key information about the flow and mixing behaviour of reaction components and thus defines the yield and selectivity of the chemical process.

Although SDR flow is theoretically characterised to be in the laminar regime [15], the film surface is covered with numerous ripples which can induce turbulence in the layers of the film beneath its surface and affect its RTD. There are some studies which indirectly indicate that flow conditions in the SDR approach a plug flow regime [5, 16, 17], but there are, as yet, no comprehensive experimental RTD data in the literature to support this claim.

The purpose of this work is to study the influence of the hydrodynamic conditions of the thin film flow and disc configurations on the RTD in order to determine the optimal experimental parameters for which near plug flow behaviour prevails on the spinning disc. Generally, the residence time distribution measurements are obtained from tracer experiments that consist

of a pulse response method whereby the injection of tracer can be done at the inlet of the reactor and the concentration – time curve can be recorded at the outlet of the reactor. In this work, we describe a simple and direct method for processing RTD signals from conductivity measurements as applied to a spinning disc reactor system. Online conductivity measurement of the response to a KCl pulse injected into the liquid feed stream introduced to the SDR is recorded and analysed. RTD parameters such as normalised variance, dispersion number and number of tanks in series were studied under various hydrodynamic and disc surface conditions. The results obtained will be useful for the accurate reactor modelling of the performance of the SDR for a range of processes such as crystallisation and polymerisations, amongst others.

2. Materials and Methods

The schematic set up for the RTD experiments is shown in Figure 1. A peristaltic pump (Watson Marlow, 505S model) was used to provide a superficial flow, and a syringe pump (New Era pumps, NE-1000) was used to inject the tracer. A conductivity probe connected to a conductivity meter was used to record the concentration of the KCl solution used as tracer in the experiments.

Distilled water and two different mixtures of glycerol and water (50 % w/w glycerol in water and 75 % w/w glycerol in water) were used as the test fluid which was continuously fed from a reservoir into the reactor at three different flow rates between 5 and 15 ml/s. Two different types of stainless steel discs were used in this study: a smooth disc (Figure 2a) and a grooved disc with eight concentric grooves (Figure 2b).

The E61M014 conductivity probe of 4 mm in diameter and 103 mm in length was placed inside the sampling shoe so that its tip was located just on top of the outlet tube in the base of the sampling shoe. The latter was held in place on a support fixed to the base of the reactor housing and was positioned near the edge of the disc so as to capture fluid thrown off the edge of the disc. When the fluid entered the shoe, it made contact with the probe before it exited the sampling shoe through the outlet tube. The set-up of the sample shoe in the reactor housing and the probe within it is shown in a photo and accompanying schematic drawing (Figure 3a and b, respectively).

A 0.4ml aliquot of a fixed concentration (0.5 M) KCl solution (Sigma–Aldrich) was rapidly injected into the system at the centre of the disc within 0.24 s to produce as close to a pulsed injection as possible using the precisely controlled syringe pump. Although it was not possible to measure the inlet trace of the pulse input into the liquid due to the mechanical

difficulties and safety aspects of inserting the probe into the thin film on the rotating disc, we are confident that our tracer introduction procedures did not compromise the formation of a sharp pulse in the liquid at the entry point. At the outlet, the conductivity versus time was measured using the conductivity probe connected to a CDM210 conductivity meter (Hach-Lange Ltd.) The data logger was started simultaneously with the pulse injection. Conductivity was measured every 200 millisecond and logged on a computer with a DaqPro 5300 data logger. All experiments were conducted with the disc temperature controlled at 20°C.

The conductivity probe was calibrated prior to its use in the RTD experiments by using a set of solutions of known KCl concentrations at a fixed conductance range of 40 mS/cm. As a linear relationship between the concentration and conductivity was established in a concentration range up to 0.5 M, the injected KCl tracer concentration was therefore set at 0.5 M. On the basis of the conductivity-concentration linear relationship, the conductivity measurements can be directly used to obtain the age distribution functions, $E(t)$ or $E(\theta)$, as well as the mean residence time τ .

3. RTD Analysis

The residence time distributions of material in the reactor is described by the $E(t)$ curve that shows the variation of tracer concentration at the exit and is given by Eq. 1 [18].

$$E(t) = \frac{C(t)}{\int_0^{\infty} C(t)dt} \quad (1)$$

It describes in a quantitative manner how much time different fluid elements have spent in the reactor. The dimensionless formulation of the residence time distribution density $E(\theta)$ (Eq. 2), where θ is the dimensionless time parameter expressed in Eq.3, is obtained from the mean residence time τ which is calculated from Eq.4. The mean residence time is a function of C_i , the measured tracer concentration at time t_i , and Δt_i which is the interval between two measurements.

$$E(\theta) = \tau E(t) \quad (2)$$

$$\theta = \frac{t_i}{\tau} \quad (3)$$

$$\tau = \frac{\sum_i t_i C_i \Delta t_i}{\sum_i C_i \Delta t_i} \quad (4)$$

The variance or second moment of the residence time distribution, σ^2 , and the normalised variance, σ_θ^2 , can be evaluated as Eq. 5 and 6 respectively.

$$\sigma^2 = \frac{\int_0^\infty t^2 C dt}{\int_0^\infty C dt} - \tau \quad (5)$$

$$\sigma_\theta^2 = \frac{\sigma^2}{\tau^2} \quad (6)$$

The variance is a measure of the “width” of a distribution and for narrow RTD distributions, a lower variance is desirable.

3.1 Radial dispersion model

Experimental data were fitted to a plug flow with radial dispersion model applicable to film flow on the SDR for which the relevant cylindrical co-ordinates are shown schematically in Figure 4. The radial dispersion model is analogous to the axial dispersion model developed for axial flow in a pipe [19, 20], the differences being the length parameters in the two systems, as depicted in Figures 5a and b for the radial flow in the SDR and axial flow in the pipe respectively. The radial dispersion model applicable to flow on the disc is described in Eq. 7 in terms of the dimensionless time θ ($=t/\tau$, where $\tau=R/u_m$ for radial film flow) and dimensionless radial distance r' ($=r/R$).

$$\frac{\partial C}{\partial \theta} = \frac{D}{u_m R} \frac{\partial^2 C}{\partial r'^2} - \frac{\partial C}{\partial r'} \quad (7)$$

where $D/u_m R$ is termed the dispersion number and the Peclet number, Pe , is the reciprocal of the vessel dispersion number i.e $Pe=u_m R/D$.

A solution to the dispersion model, subject to open–open boundary conditions, has been formulated by Levenspiel and Smith [18] for significant deviations from plug flow for flow in a pipe. The analogous solution for radial flow on the disc is given in Eq. 8 which enables the

dispersion number to be calculated from experimental measurements of the dimensionless variance.

$$\sigma_{\theta}^2 = 2 \frac{D}{u_m R} + 8 \left[\frac{D}{u_m R} \right]^2 \quad (8)$$

In this work, measurements of conductivity were converted to tracer concentrations C (mole L^{-1}). We use a commonly employed technique, which involves pulse injection of tracer followed by measurement of the tracer concentration near the edge of disc.

3.2 Tanks-in-series model

The age exit distribution (E) for N tanks-in-series under the pulse injection is determined as Eq. 9 [18]

$$E(t) = \frac{C(t)}{\int_0^{\infty} C(t) dt} = \frac{t^{N-1}}{(N-1)\tau_i^N} e^{-t/\tau_i} \quad (9)$$

Where τ_i is the mean residence time for the i -th tank. The corresponding dimensionless form of E is determined as Eq. 10 [18].

$$E(\theta) = \tau E(t) = \frac{N(N\theta)^{N-1}}{(N-1)!} e^{-N\theta} \quad (10)$$

The number of tanks-in-series can be estimated as Eq. 11 [18].

$$N = \frac{1}{\sigma_{\theta}^2} \quad (11)$$

This expression shows that the number of tanks-in-series N necessary to model the real reactor is directly related to the dimensionless variance which is determined from the RTD profile. For flow approaching plug flow regime, N should be as high as possible. In terms of the age distribution function, a narrow profile is associated with a small variance and therefore larger N . In general, for small deviations from plug flow (i.e dispersion number <0.01), $N > 50$ and the RTD becomes symmetrical and Gaussian.

4. Results and Discussion

RTD curves for film flow in the SDR are expected to vary from very narrow symmetric distribution when there is negligible radial mixing (i.e ideal plug flow conditions), to more asymmetrical and broader when significant deviations from plug flow arise. A narrow,

symmetrical RTD is characterised by a low variance or dimensionless variance (given by equations (5) and (6) respectively).

Experiments were repeated three times at randomly selected operating conditions of 600 rpm disc rotational speed and 10 ml/s flowrate for the water system on the smooth disc to examine the reproducibility of the data collected. The graph of normalised residence time distribution (Figure 6) shows almost overlapping profiles for the three repeated experiments. The maximum error in the values for the normalised variance, axial dispersion and number of tanks in series was 3%, indicating very good reproducibility of the data.

4.1 Effect of flowrate and rotational speed

The influences of disc rotational speed and flowrate on the RTD curves are presented in Figures 7 and 8 respectively.

The general observation from Figures 7a to 7c is that the lowest rotational speed of 300 rpm is associated with a slightly broader and somewhat less symmetrical distribution than the higher disc speeds. This is especially so at the higher flowrates of 10 and 15 ml/s (Figures 7 b and c). This observation is more evident from the higher dispersion numbers calculated at 300 rpm compared to the higher disc speeds, as presented in Table 1. As the disc speed increases, the dispersion number drops for flowrates of 10 and 15 ml/s. It is noteworthy that there is, however, no clearly discernible trend in these parameters at 5 ml/s for the range of disc speeds investigated. It is possible that this may be due to the film breaking down into rivulets at this lowest flowrate of 5 ml/s especially as it thins towards the edges of the disc rotating at the highest disc speed. With the disintegration of the film, the hydrodynamics would be different from those in a continuously spreading film and would not be easily defined. Indeed film breakdown at very low flowrates in the SDR has been shown to be responsible for reduced mixing capability of the film [21]. It is envisaged that the RTD which, like mixing, is also dependent on the flow dynamics can be similarly affected.

Similarly, an increase in the flowrate from 5 ml/s to 15 ml/s results in a narrower RTD (Figures 8a to c). This effect is also reflected in the lower dispersion numbers at the larger flowrates in Table 1.

The hydrodynamics of the thin film flow can be used to explain these effects of rotational speed and flowrates on the approach to plug-flow behaviour. Achieving near plug-flow behaviour is dependent on a uniform velocity profile in the direction perpendicular to the flow direction (i.e within the film thickness) and on negligible dispersion in the direction of flow. In the context of film flow on the rotating disc where bulk flow is in the radial direction, higher flowrates and disc speeds have been shown in earlier studies [22, 23] and more recently [24]

to lead to more intense ripples on the surface of the film. These ripples are generally absent at low disc speeds and low flowrates where a smooth film in laminar flow regime prevails. When film surface instabilities or waves are present, they are likely to induce turbulence within the layer underneath via the formation of turbulent ‘eddies’ (as illustrated in Figure 9) giving a more uniform velocity profile in the transverse direction as opposed to the parabolic velocity profile that characterises waveless laminar flow. Therefore, the more waves that are formed at higher disc speeds and higher flowrates, the greater the transverse mixing and the more uniform the radial velocity across a given cross-section in the film. Moreover, the higher the centrifugal force exerted on the film at higher disc speeds (where $F_{\text{centrifugal}} \propto \omega^2 r$), the lesser would be the tendency for radial dispersion away from the direction of flow, thus suppressing deviations from plug flow behaviour. Indeed Boiarkina et al. [24] have demonstrated in a striking visual study of dye mixing in a water film on a spinning disc that extremely limited radial mixing was observed even in the presence of waves under various conditions of disc speeds and water flowrates. This proves that the interaction of the waves with the film occurs more in a transverse direction rather than in the radial direction, which supports the argument of transverse mixing we have put forward above. It is also interesting to note that near plug-flow behaviour prevailed in that work even at the relatively low rotational speeds of up to 300 rpm. The RTD investigation reported herein goes a step further to demonstrate that even sharper RTD profiles can be obtained if much higher rotational speeds are employed.

Table 1. Dispersion parameters for water on smooth disc at different flowrates and rotational speeds

	Dispersion number ($=D/u_m R$)			
Flowrate (ml/s)	300 rpm	600 rpm	900 rpm	1200 rpm
5	0.0172	0.0153	0.0170	0.0172
10	0.0166	0.0143	0.0123	0.0106
15	0.0130	0.0118	0.0094	0.0087

4.2 Effect of disc surface

The RTD profile comparisons between the smooth and grooved discs are shown in Figures 10a and 10b for different sets of operating conditions. Under identical conditions of disc speed and flowrate, it is observed that the flow on the grooved disc is generally associated with an overall narrower RTD than the smooth disc at higher disc speeds as depicted in

Figure 10a and higher flowrates (Figure 10b). The profiles in Figure 10a reveal interesting flow characteristics on the grooved disc. At the lowest disc rotational speed of 300 rpm, there is a stronger tailing at the start and end of the trace for the grooved disc than for the smooth disc, although the overall profile between these extremities is narrower. The tailing is significantly reduced at higher disc speed. The initial tail is indicative of the liquid bouncing off the surface of the grooved disc and exiting the reactor much earlier than the rest of the liquid. The explanation for this phenomenon may be related to earlier work by Burns et al. [25] which established that liquid flow on a rotating disc can be divided into three distinct regions: the injection zone, the acceleration zone and the synchronised zone. The radial velocity varies significantly in each zone (as shown in Figure 11). The injection and acceleration zones are collectively termed the ‘spin-up’ zone which determines the proportion of the disc over which the liquid is not fully coupled with the disc surface. The perturbations in the flow in the spin-up zone combined with the presence of grooves can cause the accelerating liquid to be imparted with enough energy in order to bounce off the inclined surface of the groove. The flat surface of a smooth disc on the other hand would minimise this effect.

We suggest that this behaviour would be more prevalent in systems where the inertial forces of the film dominate over the viscous forces, i.e at low Ekman numbers where the Ekman number, Ek , is defined as:

$$Ek = \left(\frac{2\pi}{3}\right)^{2/3} \left(\frac{r^4 \omega \nu}{Q^2}\right)^{1/3} \quad (12)$$

Thus a combination of low disc speeds, high flowrates and low viscosity on the grooved disc would result in liquid readily bouncing off the surface.

The end tail visible in the RTD profiles for the grooved disc is representative of liquid accumulation in the grooves. This would be expected to occur at combined conditions of low disc speeds and low flowrates, conditions under which energy of the liquid may not be enough to displace it out of the depth of the groove.

Aside from the tailing regions in the RTD of the grooved disc, for which explanations have been formulated above, it is also clear that the flow profile was generally sharper than that for the smooth disc between these two extremities (Figures 10 a and b). We postulate that this effect relates to a number of considerations. Firstly, a greater number of instabilities or turbulent eddies is generated in the film as it flows across the constantly changing surface structure. These arise as a result of film detachment from the surface and its subsequent re-

attachment downstream of the groove. Indeed, several numerical and experimental studies conducted on gravity-driven laminar liquid films flowing on wavy surfaces [26, 27] have shown that induced recirculation or a vortex in the depth of the surface structure can be set up in this manner. This occurs because the film is unable to follow the surface undulations at all times. The size of the vortex formation has been shown to vary with the Reynolds number or flowrate, with an optimum flowrate, which is presumably dependent on the indentation geometry, being responsible for the largest vortex [28]. In contrast to gravity-driven flows, flow on a rotating disc would be strongly governed by the applied centrifugal force which would exert an even greater influence on the detachment of the flow from the grooved surface, hence creating more turbulent eddies in the process. Therefore the higher the disc speed, the more eddies formed in this manner. Secondly, it has also been suggested in the literature that surface textures have the potential to suppress liquid channelling or rivulet flow compared to a smooth surface under identical conditions of liquid flowrate and liquid properties [29] so that a higher wetted area is achieved. Rivulet flow as opposed to film flow would result in larger average film thicknesses which may lead to detrimental performance, especially under strictly laminar flow conditions (i.e. where any surface wave-induced turbulence is absent). Hence, on the basis of this argument, the grooved disc in our study may promote more stable film flow than the smooth disc at a given flowrate and disc speed and may only experience film breakdown at much lower flowrates than the smooth film. Of course, the extent of this effect would be dependent on the characteristics of the texture employed and further work on film breakdown on the grooved disc would be required for a better understanding of this phenomenon and confirmation of this hypothesis. And thirdly, the waves on the free surface of the film which have been extensively discussed earlier in terms of enhancing the transverse mixing in the film (section 4.1), has been shown in a previous study to be more numerous for films on the grooved disc in comparison to a smooth disc [23]. It is likely that this is directly related to the first explanation of formation of vortices which, if they are large enough in comparison to the film thickness, extend right through to the film surface. A combination of all these enhancement factors exhibited by the grooved disc would be conducive to promoting good control of the RTD.

A quantitative depiction of the influence of the grooved disc on the RTD profile is obtained by examining the number of tanks-in-series, N , which is derived from normalised variance σ_{θ}^2 of the $E(\theta)$ profile as expressed in Eq. (11). The results are illustrated in Figure 12. It is seen that the number of tanks-in-series is generally higher with the grooved disc than with the smooth disc, with more pronounced increases in N from, for instance, 55 for the smooth disc to 65 for the grooved disc at the highest disc speed and flowrate of 1200 rpm and 15 ml/s

respectively. These data reinforce the beneficial effect of higher disc speed as explained above.

4.3 Effect of feed viscosity

Figure 13a to 13d show the influence of feed viscosity on RTD at identical flowrates and rotational speed. It can be observed that the fluid viscosity is highly influential on the shape of the RTD curves. The RTD curves tend to become more asymmetrical and are broader as the viscosity of the fluid increases. This is to be expected as higher viscosities would cause the surface waves to be dampened to such an extent that smooth, laminar flow will be more prevalent [22]. The velocity gradients thus set up within the film are responsible for the greater deviation from plug-flow as observed from the RTD profiles.

The quantitative analysis of the RTD profiles in Figures 13a to 13d, based on the tanks-in-series model, is shown in Figure 14. The N values for the higher viscosity liquids are consistently lower than those for water at any given flowrate and disc speed, indicating significant deviations from plug flow behaviour on the disc.

Further analysis of the effect of the grooved disc on the flow profile of higher viscosity liquids (Figure 15) reveals some interesting features. The tailing, especially on the end of the profile, at the highest viscosity liquid (75% w/w glycerol in water) studied in this present work, is very prominent. What is more surprising is that tailing for the grooved disc no longer surpasses that of the smooth disc, as is the case for the water system. This is indicative of the liquid's inability, under conditions of higher viscosity, to bounce off from the surface of grooved disc as easily as it did at water-like viscosities (see section 4.2). The Ekman number (Eq. 12) is now higher and therefore the inertial effects are diminished. What these results suggest is that, with the increase in viscosity, the flow is no longer as chaotic as it was with water. Nevertheless, the grooved disc should still be able to induce a certain degree of instability in the high viscosity liquid by virtue of its surface texture compared to the smooth disc and this is reflected by the tighter RTD curves in Figure 15 and lower dispersion numbers, as illustrated in Table 2.

Table 2. Dispersion parameters at flowrate of 10 ml/s and rotational speed of 1200 rpm for smooth and grooved discs with different liquid viscosities

	<i>Dispersion number, $D/u_m R$</i>
--	--

Liquid medium	Smooth Disc	Grooved Disc
water	0.0106	0.0099
50% w/w glycerol/ water	0.0214	0.0182
75% w/w glycerol/ water	0.0264	0.0207

4.4. Empirical correlation

Empirical correlations describing the influence of the independent variables angular velocity ω , flow rate Q and liquid kinematic viscosity ν on the experimentally determined Peclet number have been developed for the smooth and the grooved disc using multiple linear regression analysis. In formulating the model, we assume that the Peclet number can be represented by:

$$Pe = A \omega^a Q^b \nu^c \quad (13)$$

or in linearised form:

$$\log Pe = \log A + a \cdot \log \omega + b \cdot \log Q + c \cdot \log \nu \quad (14)$$

where: ω is angular velocity of the disc (rad s^{-1}), Q is liquid feed flow rate ($\text{m}^3 \text{s}^{-1}$), ν is the kinematic viscosity of the liquid ($\text{m}^2 \text{s}^{-1}$) and A, a, b and c are regression coefficients.

The model equations generated are as follows:

$$\text{Smooth disc: } Pe = 10^{2.561} \omega^{0.188} Q^{0.371} \nu^{-0.136} \quad (R^2=0.81) \quad (15)$$

$$\text{Grooved disc: } Pe = 10^{2.765} \omega^{0.203} Q^{0.372} \nu^{-0.104} \quad (R^2=0.73) \quad (16)$$

Eq. 15 and 16 are applicable under the following conditions:

$$\begin{aligned} 30 \text{ rad s}^{-1} &\leq \omega \leq 125 \text{ rad s}^{-1} \\ 5 \times 10^{-6} \text{ m}^3 \text{s}^{-1} &\leq Q \leq 15 \times 10^{-6} \text{ m}^3 \text{s}^{-1} \\ 1 \times 10^{-6} \text{ m}^2 \text{s}^{-1} &\leq \nu \leq 3 \times 10^{-5} \text{ m}^2 \text{s}^{-1} \end{aligned}$$

The empirical model equations confirm that increases in disc rotational speed and liquid flow rate and a decrease in viscosity lead to higher Peclet number and therefore near plug flow

behaviour for both disc surfaces. Overall, the flow rate Q is predicted to be the more influential parameter than the disc speed, although the grooved disc exhibits a stronger dependency on the disc speed than the smooth disc. Similarly, the viscosity effect seems to be less important in the presence of grooves. These predicted trends are in agreement with our experimental observations.

The reasonably good fit between the experimental data and model prediction is illustrated in Figures 16 a and b for smooth and grooved disc respectively. There is clearly less agreement between the model predictions and the experimental data at the highest Pe numbers tested in this work ($Pe > 100$). It would be beneficial to generate more experimental data in this range of Pe, i.e at higher disc speeds and flow rates to improve the predictive models obtained for both the grooved and smooth discs.

5. Conclusions

With increases in the disc rotating speed and the liquid flow rates, the RTD of the thin film flowing on the rotating disc becomes narrower. Quantitatively, the rotational speed effect is reflected in the reduction in the radial dispersion number ($=D/u_m R$) by more than 30% with an increase in disc speed from 300 to 1200 rpm, at a constant flow rate of 15 ml/s, indicating that the flow behaviour approaches that in an ideal plug flow reactor. At higher disc speeds, the centrifugal force pulling the liquid film to the edge of the disc is greater, which may be responsible for the suppression of any tendency for dispersion in the radial direction. Also, it is suggested that greater transverse mixing induced by more numerous surface waves at higher disc speed contributes to equalising the velocity profile at any given cross section of the film. Similarly, the dispersion number decreases by almost 50% as flow rate is increased from 5 ml/s to 15 ml/s. This observation can again be explained by the higher velocity and the turbulence induced by the ripples on the fluid film. Using a textured surface such as concentric grooves promotes narrower RTD of the water and high viscosity liquids compared with the smooth disc. The highest number of tanks in series is estimated as 65 for the grooved disc compared to 55 for the smooth disc at identical conditions of 1200 rpm rotational speed and 15 ml/s flow rate, with water as the working fluid. The observations of tailing at the beginning and end of the RTD profile with water on the grooved disc has been explained in terms of distinctive hydrodynamic conditions which are applicable to the water film only. Another key point that can be gleaned from these results is that, in order to partly overcome the detrimental effect of viscosity on flow behaviour, changing the disc surface from smooth to grooved is a feasible alternative. The effects on the RTD of all the

parameters investigated in this study have been validated by regression models for the Peclet number developed for both the smooth and grooved discs.

Overall, this investigation provides a valuable insight into the operating conditions of the SDR which generate flow profiles approaching plug flow behaviour. The results will help inform the choice of operating parameters for reactions conducted on the spinning disc which are most sensitive to macromixing effects (e.g competitive reaction schemes, crystallisations) in order to maximise yield and selectivity and control product properties.

Acknowledgements

The authors are grateful to Dr Anh Phan (Newcastle University) for the loan of the conductivity probe used in this study and for her valuable guidance in using the equipment.

Nomenclature

$E(t)$	Age exit distribution
$E(\theta)$	Dimensionless age exit distribution
C_i	Measured tracer concentration at time t_i [mol L^{-1} or kmol.m^{-3}]
C	Tracer concentration [mol L^{-1} or kmol.m^{-3}]
D	Dispersion coefficient [$\text{m}^2 \text{s}^{-1}$]
Ek	Ekman number
L	Characteristic length scale for flow in pipes [m]
N	Number of tanks in series
Pe	Peclet number
Q	Liquid flowrate [$\text{m}^3 \text{s}^{-1}$]
r	Radial position from disc centre [m]
r'	Dimensionless radius ($=r/R$)

R	Disc radius [m]
Re	Reynolds number
t	Time [s]
t_i	Conductivity measurement time [s]
Δt_i	Time interval between two conductivity measurements [s]
u_m	Mean radial velocity across disc surface [m.s^{-1}]
z	Vertical distance along the z-axis [m]

Greek alphabets

ν	Kinematic viscosity of fluid [m^2s^{-1}]
θ	Dimensionless time ($=t/\tau$ or t_i/τ)
φ	Angular direction
σ	Variance [s^2]
σ_θ	Dimensionless variance
τ	Mean residence time [s]
ω	Angular velocity [rad.s^{-1}]

References

1. Boodhoo, K.V.K. and R.J. Jachuck, *Process intensification: spinning disk reactor for styrene polymerisation*. Applied Thermal Engineering, 2000. **20**(12): p. 1127-1146.
2. Boodhoo, K.V.K., W.A.E. Dunk, and R.J. Jachuck, *Influence of centrifugal field on free-radical polymerization kinetics*. Journal of Applied Polymer Science, 2002. **85**(11): p. 2283-2286.

3. Oxley, P., et al., *Evaluation of Spinning Disk Reactor Technology for the Manufacture of Pharmaceuticals*. Industrial & Engineering Chemistry Research, 2000. **39**(7): p. 2175-2182.
4. Matar, O.K. and C.J. Lawrence, *The flow of a thin conducting film over a spinning disc in the presence of an electric field*. Chemical Engineering Science, 2006. **61**(12): p. 3838-3849.
5. Leveson, P., W.A.E. Dunk, and R.J. Jachuck, *Investigation of shear effects on styrene free radical polymerization using a narrow channel reactor*. Journal of Applied Polymer Science, 2004. **94**(4): p. 1365-1369.
6. Moghbeli, M.R., S. Mohammadi, and S.M. Alavi, *Bulk free-radical polymerization of styrene on a spinning disc reactor*. Journal of Applied Polymer Science, 2009. **113**(2): p. 709-715.
7. Boodhoo, K.V.K., W.A.E. Dunk, and R.J. Jachuck, *Continuous photopolymerization in a novel thin film spinning disc reactor*. Photoinitiated Polymerization, 2003. **847**: p. 437-450.
8. Boodhoo, K.V.K., W.A.E. Dunk, and R.J.J. Jachuck, *Spinning disc reactor for photopolymerization*. Abstracts of Papers of the American Chemical Society, 2001. **222**: p. 520-POLY.
9. Boodhoo, K.V.K., et al., *Classical cationic polymerization of styrene in a spinning disc reactor using silica-supported BF₃ catalyst*. Journal of Applied Polymer Science, 2006. **101**(1): p. 8-19.
10. Brechtelsbauer, C., et al., *Evaluation of a spinning disc reactor for continuous processing*. Organic Process Research & Development, 2001. **5**(1): p. 65-68.
11. Alvarez, A.J. and A.S. Myerson, *Continuous Plug Flow Crystallization of Pharmaceutical Compounds*. Crystal Growth & Design, 2010. **10**(5): p. 2219-2228.
12. Chen, J., et al., *Pharmaceutical Crystallization*. Crystal Growth & Design, 2011. **11**(4): p. 887-895.
13. Sha, Z. and S. Palosaari, *Mixing and crystallization in suspensions*. Chemical Engineering Science, 2000. **55**(10): p. 1797-1806.
14. Gutierrez, C.G.C.C., E.F.T.S. Dias, and J.A.W. Gut, *Investigation of the residence time distribution in a plate heat exchanger with series and parallel arrangements using a non-ideal tracer detection technique*. Applied Thermal Engineering, 2011. **31**(10): p. 1725-1733.
15. Burns, J.R. and R.J.J. Jachuck, *Determination of liquid–solid mass transfer coefficients for a spinning disc reactor using a limiting current technique*. International Journal of Heat and Mass Transfer, 2005. **48**(12): p. 2540-2547.
16. Chin, S.F., et al., *Size selective synthesis of superparamagnetic nanoparticles in thin fluids under continuous flow conditions*. Advanced Functional Materials, 2008. **18**(6): p. 922-927.
17. Tejas J. BHATELIA, R.P.U., Vishnu K. PAREEK and Moses O. TADE1, *Characterizing liquid film thickness in Spinning Disc Reactors*, in *Seventh International Conference on CFD in the Minerals and Process Industries CSIRO2009*: Melbourne, Australia.
18. Levenspiel, O., *Chemical reaction engineering*. Vol. 3rd Edition. 1999, New York: Wiley.
19. Danckwerts, P.V., *Continuous flow systems: Distribution of residence times*. Chemical Engineering Science, 1953. **2**(1): p. 1-13.
20. Levenspiel, O., *Tracer Technology: Modeling the Flow of Fluids* 1957: Springer.
21. Boodhoo, K.V.K. and S.R. Al-Hengari, *Micromixing Characteristics in a Small-Scale Spinning Disk Reactor*. Chemical Engineering & Technology, 2012. **35**(7): p. 1229-1237.

22. Aoune, A. and C. Ramshaw, *Process intensification: heat and mass transfer characteristics of liquid films on rotating discs*. International Journal of Heat and Mass Transfer, 1999. **42**(14): p. 2543-2556.
23. Jachuck, R.J.J. and C. Ramshaw, *Process intensification: Heat transfer characteristics of tailored rotating surfaces*. Heat Recovery Systems and CHP, 1994. **14**(5): p. 475-491.
24. Boiarkina, I., S. Pedron, and D.A. Patterson, *An experimental and modelling investigation of the effect of the flow regime on the photocatalytic degradation of methylene blue on a thin film coated ultraviolet irradiated spinning disc reactor*. Applied Catalysis B: Environmental, 2011. **110**(0): p. 14-24.
25. Burns, J.R., C. Ramshaw, and R.J. Jachuck, *Measurement of liquid film thickness and the determination of spin-up radius on a rotating disc using an electrical resistance technique*. Chemical Engineering Science, 2003. **58**(11): p. 2245-2253.
26. Negny, S., M. Meyer, and M. Prevost, *Study of a laminar falling film flowing over a wavy wall column: Part II. Experimental validation of hydrodynamic model*. International Journal of Heat and Mass Transfer, 2001. **44**(11): p. 2147-2154.
27. Negny, S., M. Meyer, and M. Prevost, *Study of a laminar falling film flowing over a wavy wall column: Part I. Numerical investigation of the flow pattern and the coupled heat and mass transfer*. International Journal of Heat and Mass Transfer, 2001. **44**(11): p. 2137-2146.
28. Heining, C., T. Pollak, and N. Aksel, *Pattern formation and mixing in three-dimensional film flow*. Physics of Fluids, 2012. **24**(4): p. 042102-16.
29. Iso, Y. and X. Chen, *Flow transition behavior of the wetting flow between the film flow and rivulet flow on an inclined wall*. Journal of Fluids Engineering, Transactions of the ASME, 2011. **133**(9).

Figure 1. Experimental setup for the determination of the RTD of the SDR

Figure 2. (a) Smooth disc surface (b) Grooved disc surface

Figure 3. (a) Set-up of probe and sampling shoe in reactor housing (b) Schematic of the set-up showing probe location and positioning in sampling shoe

Figure 4. Cylindrical co-ordinates for film flow in SDR

Figure 5(a) Side view of SDR film flow in radial direction on disc of radius R ; (b) Side view of axial liquid flow in pipe of length L

Figure 6. Reproducibility of experiments at 600 rpm disc speed and 10 ml/s flow rate of water on smooth disc

Figure 7. Influence of rotational speed on RTD on smooth disc, water feed conditions, (a) 5 ml/s; (b) 10 ml/s (c) 15 ml/s

Figure 8. Effect of flowrate on RTD on smooth disc, water system and (a) 300 rpm; (b) 900 rpm; (c) 1200 rpm

Figure 9. Flow of liquid film on rotating disc (a) smooth film (b) turbulent film

Figure 10. Influence of grooved disc on RTD curves with water feed (a) 300 rpm and 1200 rpm at flowrate of 10ml/s (b) 5ml/s, 15 ml/s at rotational speed of 1200 rpm

Figure 11. Various 'zones' of operation on rotating disc, with spin-up zone represented by combined injection and acceleration zones [25]

Figure 12. Number of tanks-in-series for smooth disc vs. grooved disc at 300 rpm and 1200 rpm disc rotational speed (water feed conditions)

Figure 13. Effect of feed viscosity on RTD on smooth disc, 10 ml/s and (a) 300 rpm; (b) 600 rpm; (c) 900 rpm; (d) 1200 rpm

Figure 14. Effect of flowrate and speed on number of tanks-in-series (water, 50% w/w glycerol and water and 75% w/w glycerol and water working fluid)

Figure 15. Effect of disc surface and fluid viscosity on RTD (rotational speed of 1200 rpm and flowrate of 10 ml/s)

Figure 16. Comparison of experimental data and predicted data from empirical model of Pe for (a) smooth disc (b) grooved disc