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Optimisation of stirred vessel geometry for the drawdown and incorporation of floating solids to prepare concentrated slurries

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ABSTRACT

This paper reports on a Design of Experiments (DoE) approach to optimise the geometric configuration for effective drawdown and incorporation of floating solids to prepare high solid content slurries. The impeller speed and power draw required to ensure all dry powder is incorporated within four seconds of addition to the vessel free surface, N_{ji} and P_{ji} , were used as metrics to determine incorporation performance. Mixed flow pitched blade turbines at $D/T = 0.5$ were used. The main parameters considered were the impeller pumping direction (up versus down), impeller submergence, eccentricity, and angle of tilt. DoE was used to examine both the independent effects of the main parameters and their interactions.

Pumping mode was found to be the most significant parameter, with down-pumping impellers generally providing the best drawdown and incorporation performance. This is related to the strong interaction between pumping mode and all other parameters, where adding tilt or eccentricity reduced drawdown performance for up-pumping impellers, yet caused improvement in the case of down-pumping impellers.

The optimal geometry from the DoE was found using a down-pumping PBT, 10° tilt, 10% of the vessel diameter eccentricity and placed at an initial submergence of half the liquid height. This geometry is shown to reduce the time required to prepare a 50 wt% slurry by two thirds compared to a generic Rushton turbine design, emphasising the benefits of rational impeller and vessel design.

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1. Introduction

Drawdown of floating solids in stirred vessels is a common process operation for many industries to incorporate solids for dissolution, reaction, or suspension and slurry preparation. Examples of drawdown processes can be found throughout the polymer, paint, food, and catalyst industries, amongst others. The specific requirement of the drawdown duty is highly dependent upon the solid and liquid phase composition. For example the drawdown, incorporation and suspension of small particle ceramic materials in concentrated slurries for paints or catalyst washcoats will behave differently to the drawdown of

low solid concentration buoyant particles for dissolution, mass transfer, ion exchange, or reaction processes (Siddiqui, 1993).

Solid particles may float for a variety of reasons (Waghmare et al., 2011). Firstly, if the density of the solid particles is lower than the fluid they will float if not agitated. Using agitation to draw these particles down into the fluid forms a dynamic equilibrium where, if agitation ceases, they will return to rest at the top surface. Secondly, if the interfacial tension between the solid and liquid is sufficiently high, this causes a force at the surface with a larger magnitude than the gravitational settling force which prevents the particles from sinking, even if they have a higher density than the fluid (Rouquerol et al., 2013). Thirdly, particles

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Nomenclature

| | |
|----------|------------------------------------|
| α | Impeller tilt angle ($^{\circ}$) |
| C | Impeller off-bottom clearance (m) |
| D | Impeller diameter (m) |
| E | Impeller eccentricity (m) |
| H | Liquid height (m) |
| H_0 | Initial liquid height (m) |
| N | Impeller speed (RPM) |
| P | Impeller power draw (W) |
| S | Impeller submergence (m) |
| S_0 | Initial impeller submergence (m) |
| T | Vessel diameter (m) |

can agglomerate at the surface, with liquid bridges between particles, forming a large semi-wet mass with occluded air. The presence of this air gives this agglomerate a lower envelope density than the original particles and so it may float until it is broken up. An important distinction between the three cases is that whilst the first is reversible, the latter two are not. Once the particles are either pulled through the surface and/or fully wetted they will become non-buoyant and will not return to the surface once agitation is stopped; rather they will most commonly sediment.

Each of the three phenomenon described above do not necessarily happen in isolation. For example, in the case of porous ceramic powders all can potentially occur. Initially, the pores of the particles are filled with air and so the envelope density will be low. As the pores fill with fluid (a process that depends on the interfacial tension between the two phases) the envelope density will increase until it rises to above that of the fluid. However, the particles may also agglomerate as they hit the liquid surface, leading to a very complex force balance on the system.

Due to the complexity of the problem, previous studies have largely focused on simple systems. For example, large, low density buoyant particles have been used to isolate the phenomena (Hemrajani, 1988; Khazam and Kresta, 2008, 2009; Özcan-Taşkin, 2006). The effect of various geometric parameters on the impeller speed (N_{JD}) and power (P_{JD}) to just drawdown the solid from the liquid surface have been explored. The just drawdown criterion, N_{JD} , first proposed by Joosten et al. (1977), is the impeller rotation rate at which no solid spends more than four seconds at the free surface. This is analogous to the well-known “just suspended” criterion by Zwietering (1958) which is the impeller rotation rate at which no particle spends more than 2 s in contact with the vessel bottom.

Whilst N_{JD} is a useful parameter to study the effect of geometry at a given solids concentration, it relies upon the reversibility of the drawdown process. This is of course only true for the first of the above three conditions given by Waghmare et al. (2011). In the context of the present study, all three apply and hence the drawdown process is not reversible. Therefore a similar condition, the “just incorporation” condition, was proposed by Wood et al. (2018) for non-buoyant solids that can be incorporated into slurries. This measurement is very similar, measuring the impeller speed required, N_{JI} , to ensure all powder added is drawn down and incorporated within four seconds of addition, where a fixed amount of solid is added at a time at a fixed frequency, allowing measurement of drawdown to be carried out for concentrated systems.

Amongst previous studies there is a general consensus that mixed flow pitched blade turbine (PBT) impellers give the best performance, with significant power and speed savings compared to radial flow impellers (Joosten et al., 1977; Khazam and Kresta, 2009; Özcan-Taşkin, 2006; Özcan-Taşkin and McGrath, 2001; Özcan-Taşkin and Wei, 2003; Takahashi and Sasaki, 1999; Wood et al., 2018). The majority of these works have focussed on down-pumping impellers, although Özcan-Taşkin and Wei (2003) demonstrated that up-pumping impellers ran at lower N_{JD} and P_{JD} than down-pumping impellers when placed close to the surface. Given the consistent conclusions within previous litera-

ture, only pitched blade turbines are considered in this study; both up- and down-pumping.

The effect of submergence on drawdown performance has been considered by several researchers, with conflicting conclusions. Özcan-Taşkin and McGrath (2001) reported good performance at high impeller submergences, specifically for radial flow impellers and downward pumping PBTs. Khazam and Kresta (2009) showed that the cloud depth within the vessel improved with a higher submergence at the cost of increasing both the impeller speed and power required for drawdown for a novel geometry using a down-pumping impeller, regardless of baffle configuration. Özcan-Taşkin and Wei (2003) showed that whilst drawdown performance, in terms of N_{JD} , improved as the submergence was increased for down-pumping impellers, the opposite was true for up-pumping impellers. Khazam and Kresta (2009) made a similar observation that up-pumping impellers are much more sensitive to the effect of submergence than down-pumping impellers.

Previous studies focussing on low solid contents and solids that cannot be incorporated demonstrated an improvement in drawdown performance when using baffles. Various baffle geometries have been studied and generally show improved performance over the unbaffled case; this includes the use of one, two and four baffles that can either be full vessel height or surface only baffles (Hemrajani, 1988; Karcz and Mackiewicz, 2009; Khazam and Kresta, 2009; Özcan-Taşkin and McGrath, 2001; Siddiqui, 1993). However, baffles have been shown to inhibit the drawdown and incorporation of incorporable solids (i.e. those that, once wetted, incorporate to form a slurry rather than returning to the surface) during slurry preparation, especially as the slurry solid content is increased above 10% (Wood et al., 2018). Therefore, it is important to examine non-standard geometries to reduce quasi-solid body rotation and improve mixing performance within the vessel. The use of eccentric impellers is a common technique to improve mixing in unbaffled systems, shown to give equally efficient mixing as a baffled vessel (Hall et al., 2004, 2005). Waghmare et al. (2011) demonstrated some promise in the use of a tilted impeller for drawdown, a practice that has been shown to potentially improve mixing performance over unbaffled systems (Chung, 2008).

There are a limited number of studies that consider the effect of increasing the concentration of the solid phase on the mixing system. Xie et al. (2007) studied the deagglomeration of fumed silica agglomerates of up to 10 wt% and found that the drawdown requirement (in terms of drawdown time) increased exponentially with increasing solid concentration for all impellers studied. Khazam and Kresta (2009) examined a system containing expanded polystyrene up to a maximum concentration of 10% by volume and found the drawdown requirements (in terms of N_{JD}) significantly increased with increasing solid concentration. Özcan-Taşkin (2012) studied the incorporation of nanoscale clusters into a suspension using a proprietary design of mixer and found that the drawdown requirement (in terms of incorporation time) increased with increasing solid concentrations, especially above solid concentrations of 10 wt% up to a maximum of 20 wt%.

The effect of D/T is significant on drawdown performance and has been studied by multiple authors (Joosten et al., 1977; Özcan-Taşkin and McGrath, 2001; Özcan-Taşkin and Wei, 2003; Takahashi and Sasaki, 1999), again with varying conclusions. Generally larger impellers require lower speeds to achieve the same drawdown performance at the cost of increased power draw. However, Wood et al. (2018) demonstrated that a larger diameter PBT ($D/T = 0.5$) provided much better incorporation at higher solids content (>40%) and this outweighed the lower power of smaller D/T at low solids content (<20%) in overall process terms.

Design of Experiments (DoE) is a useful tool to ensure the maximum information is obtained from a process using a minimised set of experimental conditions. In a factorial DoE approach, process parameters are varied systematically within an orthogonal design space in order to assess efficiently the effect of each considered parameter on an output, or response variable. This approach also allows the consideration of interactions between the process variables while minimising aliasing between them, allowing optimisation of that response for a given system (Montgomery, 2012).

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