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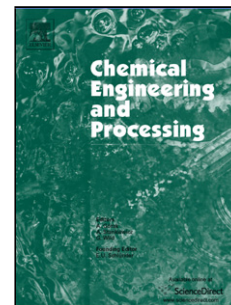
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Manuscript

## Process control in intensified continuous solids handling

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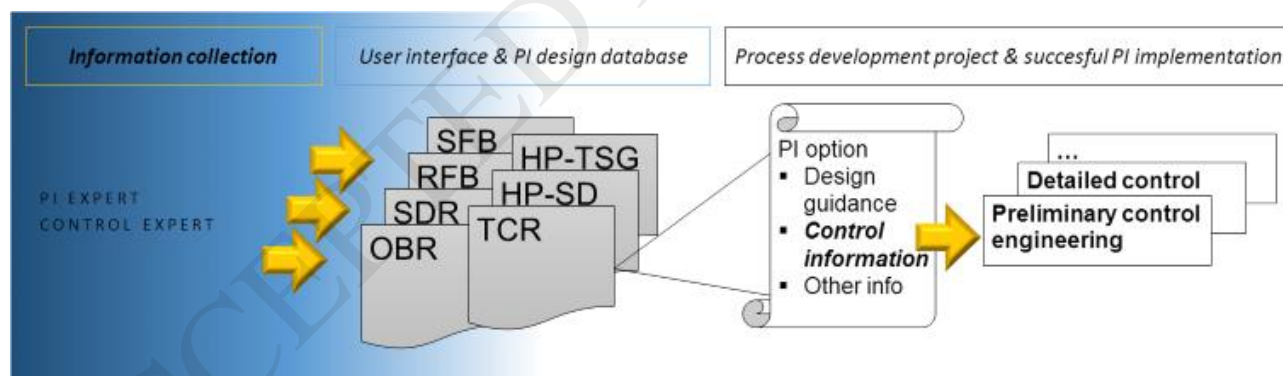
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Graphical abstract



## Highlights

- Process intensification makes strong demands on process control
- Control-oriented studies for PI involving solids are almost non-existent
- Methodology to integrate the PI and process control design is presented
- Process control discussed for eight PI technologies targeted to solids handling
- Most PI technologies are amenable to model-based control design approach

## Abstract

The application of process intensification (PI) techniques in solids handling processes requires careful assessment of challenges and limitations set by the solid phase present in the process streams. Preferably, the PI implementation involves a holistic way of thinking that covers all necessary aspects during the design phase. One of the key requirements for successful PI application is a feasible process control design that enables one to operate the process at its designed operation point. In this study, the early stage control considerations are presented for a selection of PI technologies targeted for continuous solids handling processes. The information collected in this work can be linked to the design flowsheet of each PI and is therefore readily available for a process development team to facilitate integrated process and control design. The methodology presented can be used to diminish the gap between PI and control for any PI technology.

Keywords: Process intensification; process control; monitoring; particle technology; model-based approach; integrated design

## 1 Introduction

Process intensification (PI) is traditionally understood as process development leading to a reduction in equipment size. The modern interpretation of PI extends to benefits related to business, process, and environmental aspects [1,2]. Successful applications of PI can be found in chemical engineering, such as miniaturized reactors, fuel processing systems, power sources, and integrated unit operations (e.g., reactive distillation and dividing-wall columns) [3–5]. In solids handling processes in pharmaceutical, ceramic, and mineral processing industries, for example, the application of PI requires careful assessment of challenges and limitations set by the solid phase present in the process streams [2].

It has been recognized that PI implementation could benefit from a holistic (global) way of thinking in order to meet the process development timeline demands (see [5] and references therein). Such an approach for the solids handling processes has recently been taken in the Intensified by Design project<sup>1</sup>. According to Law et al. [6], the key requirement for applying PI to a given solid handling process is to have a full understanding of the process involved. This can be broken down into four features: (1) Propensity for fouling/scaling/blockage, (2) reaction kinetics/rates, (3) full solubility/equilibrium data, and (4) the proposed flowsheet with all the unit operations involved. Here, we propose to add another feature to this holistic framework for successful PI application—the requirements of process control and monitoring. Indeed, the theoretical increment in process efficiency gained through any PI application might be compromised if the plant is difficult to control and therefore cannot be operated at its nominal operating point [7]. Traditionally, process control design has been conducted separately after the process design and usually by other experts. This kind of sequential approach simplifies the overall process synthesis and is easy to understand from the management and resources point of view, but on the other hand, it means that the control design problem is constrained by the process design decisions [8]. With the integrated process and control design, such bottlenecks can be efficiently avoided. Methods for the integrated design have been reviewed, for example, in [9].

Process control and monitoring issues in PI processes have received a fairly limited amount of attention, although the challenges have been recognized. For example, Nikačević et al. [10] mentioned the limited actuation possibilities, propagation of disturbances, nonlinear behavior and narrow operating and actuation ranges. In addition, PI processes typically involve faster and more complex dynamics (response times) [3], and it is suggested that in intensified processes, the dynamics of sensors and actuators may also play a crucial role in controller design and control performance [11,12]. In general, the higher degree of integration will make the process more challenging to control and will perhaps restrict the implementation of highly intensified processes in industry [7]. Many of the published works on control design for intensified processes deal with reactive distillation (see, e.g., [10,13–15]), but reported work in the area of solids

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<sup>1</sup> <http://ibd-project.eu/>

handling or particulate processes is even more rare. Vangsgaard et al. [16] presented a process-oriented approach to controller design for a novel, intensified single-stage autotrophic nitrogen removing granular sludge bioreactor (CANR). Su et al. [17] investigated how existing batch crystallization operation and its control technique could be converted into continuous mode. Ghiasy et al. [18,19] have studied the control strategies of a spinning disc reactor applied to an acid-base neutralization process and the reactive precipitation of barium sulfate. Bahroun et al. [20] proposed a two-layer hierarchical control approach for an intensified three-phase catalytic slurry reactor. In [21], nonlinear model predictive control (NMPC) approaches were applied for an intensified continuous hydrogenation reactor.

In general, solids handling and particulate processes present a difficult control problem characterized by the dispersed solid phase and the continuous fluid-based medium. Although population balance models (PBM) can be derived to describe the governing nonlinear phenomena and complex dynamics [22], the traditional process control solutions are typically targeted to linear systems. In addition, the heterogeneity of the processed material poses severe sampling and monitoring challenges. While operation of conventional processes can mainly rely on standard process measurements and analyzers using samples extracted from the process, the increased speed of process response times in PI processes may often require fast and reliable, nonintrusive in-line measurements. Process analytical technology (PAT) offers several interesting monitoring and control solutions for particulate and PI processes [23]. Advanced control and intelligent methods offer a variety of tools for coping with uncertain, nonlinear, and time-varying processes; for optimizing the operation; and for replacing difficult measurements with inferred measurements [24–26]. Examples can be found not only from industrial processes but also in automotive applications [27,28], electrical engineering [29], and robotics [30]. With the additional challenges arising from PI, a successful process design project involving PI and solids handling requires taking the process monitoring and control aspect into consideration at an early stage. It is crucial to identify monitored variables (including controlled variables and disturbances) and the level of monitoring needed, to select which of the monitored variables needs to be connected to closed-loop control, and to identify the available manipulated (correcting) variables. In addition, availability of mathematical models enables the

evaluation of whether the process can be kept in its optimized operation point with the available manipulated variables and then be used as a basis for advanced process control.

In this study, these early stage control considerations are applied to a selection of PI technologies targeted for continuous solids handling processes. The information collected can be linked to the design flowsheet of each PI to make it readily available for a process development team. With the information provided, the integrated process and control design is inherently initialized, and the intensified process design will more likely have a fit-for-purpose and intensified process monitoring and control solution.

This article begins by presenting the studied technologies and the information collection methodology in the Material and Methods section, followed by the Results and Discussion section, which presents the gathered findings for each PI technology, summarizes the control design readiness for the studied PI technologies, and discusses other related issues between PI and process control. Final remarks are given in the Conclusions section.

## 2 Material and Methods

### 2.1 Studied technologies

The studied PI technologies and potential applications are presented in Table 1. The information concerning the potential applications shown in Table 1 is collected from [2], where the available PI technologies for solid handling applications have been reviewed. For each studied technology, qualitative information for control and monitoring issues has been formulated. The procedure for the information collection is presented in the following section. As an inherent part of the information collection, short descriptions of the PI technologies are generated and presented in the Results and Discussion section. Further details on the studied technologies can be found, for example, from [2] and the literature cited in the Results and Discussion section.

## 2.2 Information collection

Formulation of the qualitative information for control and monitoring issues allows inspection of the list of candidate variables for process control, study of the known or expected interactions between these variables, and determination of preliminary control configurations. The workflow for collecting the information for the studied PI technologies is described in Figure 1. First, a control questionnaire based on the systematic procedure presented in [31] was prepared. The questionnaire can be found in Appendix. The questionnaire was completed by the PI experts and includes the relevant literature sources both from the PI and control points of view. As a result, the possible controlled variables (CV), manipulated variables (MV), and disturbance variables (DV) or observable variables (OV) for each PI technology were defined and listed. The variables could be related to mechanical or hydrodynamic performance of the PI equipment, or they can be related directly to potential process applications. Next, the variables were incorporated in an interaction table indicating the known or expected magnitude (steady state) and speed (dynamic) of interaction between the variables. Finally, general findings considering the different control strategies were made, and the availability of model-based tools was addressed.

As indicated in Figure 1, the control information can be implemented, for example, into a PI design database. As shown in Figure 2, a process development team can access and use this information as part of their design process. The control design subtasks illustrated in Figure 2 are based on the systematic framework given in [32]. The control information embedded in the PI design database supports the control concept development stage. Due to the high number of different applications for each PI, as indicated in Table 1, the information collection produces a limited number of potential CVs and DVs to be considered in the control concept development stage, rather than a comprehensive list with all possible application-related variables. The final decision on CVs and therefore the control objectives requires application-dependent process knowledge.

### 2.3 Interaction table

Essentially, the interaction table is an easily accessible tool to present the variable candidates to be incorporated in process monitoring and control, to evaluate the complexity of interactions, and to assess the requirements for the process monitoring and control solutions while considering a selected PI technology to a given solid handling process. An example of an interaction table is presented in Table 2. In the table, the magnitude of the known interaction is indicated as Large, Moderate, or Small. The dynamic response of the interaction is given as Fast, Fair, or Slow. Both scales are based on the largest/fastest interaction if not stated otherwise. In the subsequent process design stages, the qualitative information can be replaced with the quantitative data for a steady-state process model for control design [33].

From Table 2, it can be observed that the PI technology may need to be accompanied by monitoring solutions for detecting product particle size and moisture (application-dependent CVs). Flow regime (hydrodynamic CV) is affected by several MVs and one DV, suggesting the need for model-based inspection of interactions during the process design. Minimization of disturbances arising from feed particle density should be considered in the process design, or the feed particle size should be measured and compensated for in the control design as it disturbs both of the listed product properties. It is also clear that the PI equipment offers a limited number of MVs with interactions to a number of CV candidates. This indicates that multivariable control strategies should be preferred. Advanced process control may be required to find the optimized combination of set points for the MVs to fulfill the quality targets of multiple CVs. If the power consumption is also treated as a CV, the control problem becomes even more challenging.

## 3 Results and Discussion

For each studied PI technology, the interaction table, the control findings made based on the control questionnaire, and the literature sources are presented in the following subsections. Finally, the lessons learned from these exercises are summarized, and other aspects necessitating further study are discussed.



### 3.1 OBR

The oscillatory baffled reactor (OBR) is a tubular reactor fitted with equally spaced baffle plates. Either the fluid or the baffles are oscillated to improve the mixing performance and maintain a plug flow behavior. The OBR is suitable for continuous operation with long reaction times. Conventional OBRs have diameters higher than 15 mm, and mesoscale OBRs have diameters less than 5 mm. Design and operation aspects of OBRs are well described in [34], and a more detailed review is given in [35]. Mesoscale OBRs have been studied recently, and a review [36] and several experimental works [37,38] have been published. OBRs have been used in crystallization [35], suspension polymerization [39], and bioprocesses involving microalgae cultivation [40].

The interaction table for the OBR is presented in Table 3. Manipulated variables in OBRs are the feed flow rates, oscillation frequency and amplitude, and temperature. It has been shown that product quality attributes, such as mean particle size and particle size distribution (PSD), can be controlled by oscillation conditions while keeping polymer chemistry the same [41]. The study in [39] showed that stable dispersion conditions cannot be achieved with any combination of the oscillation amplitude and pulsation frequency. Evidently, manipulation of the oscillation conditions has a direct effect on fluid dynamics and therefore on product quality attributes. Therefore, changes in oscillation conditions require a multivariable control approach, where the interactions are accounted for or introduced as constraints. Process constraints may also arise from operating pressure and throughput to avoid particle sedimentation. Temperature control may involve temperature profile control, where different sections of the OBR should be adjusted to different processing temperatures.

Modeling of the OBR is at a mature stage: Ni et al. [41] have developed population balance rate equations for a conventional OBR. The residence time distribution of mesoscale OBRs have been described with tanks-in-series models [38]. Numerical simulations of OBR have also been performed, for example, in [42,43]. These studies could be used as a basis for model-based control, especially if a variety of products

need to be produced, or process disturbances affecting product quality are found and need to be attenuated.

### 3.2 SDR

The spinning disc reactor (SDR) provides fast mixing, mass and heat transfer rates, which can be usefully exploited in, for example, nanoparticle precipitation [44–46], polymerization processes [47,48] and organometallic processes in the pharmaceutical industry [49], among others. The process involves a rotating disc with controllable speed and temperature. Reagents are typically fed onto the center of the disc, where they form a thin film. Reactive or inert gases can also be delivered over the thin film. The reactor walls may also be temperature controlled, and the reactor pressure may be regulated.

According to the interaction table presented in Table 4, temperature, rotational speed, and reagent feed flow rates can be adjusted in SDR operation. The fluid dynamic parameters (film thickness, disc residence time, and shear rate) affecting application-dependent product quality attributes (e.g., particle size and pH) are all strongly interacted by manipulations in rotational speed and flow rate. Temperature has a smaller influence on fluid dynamics, but it has a strong effect on reactions rates, conversion, and yield. The process constraints are generated from the cut-off point of disc speed and feed flow rate adjustments because there is a trade-off between residence time and mixing performance [48,49]. Additionally, the reagent concentrations (ratio of reagents) have a strong effect on the operating windows and dynamics, as they determine whether the system is residence time controlled (kinetically limited) or mixing intensity controlled (mixing limited). Therefore, maximizing yield and optimizing product quality require balancing between rotational speed and flow rates [46]. Particle accumulation can disturb the process measurements, but it may also disturb the particle growth mechanisms on the disc. If obvious process disturbances are not expected, and the SDR shows robust performance, the control problem basically involves a safe start-up and maintaining the processing conditions at their set points. Different products could then be produced based on different recipes incorporating predetermined reagent concentrations, flow rates, and rotational speeds. On the other hand, if advanced online control for SDR is required, the

control problem is challenging due to nonlinear characteristics, fast dynamics, short residence times, and high responsiveness, as shown in [19]. The measurement and transport delays with typical instrumentation are much greater than the dynamics of SDR, and they limit the control performance.

The hydrodynamics of the SDR in nanoparticle production using Computational Fluid Dynamics (CFD) has been described, for example, in [44]. In their later study, de Caprariis et al. [50] used the developed CFD model with population balances to predict crystallite dimensions. Ghiasy et al. [18,19] have used sensors and actuators available on the market and linear control strategies for SDR in their experiments, but they also expressed mathematical relations between the disc rotational speed and the micromixing time constant (affecting the rate of precipitation), as well as the disc speed and the residence time in their experimental conditions. Additionally, the effects of several operating parameters on product size distribution and yield in a crystallization process have been systematically studied [46]. These studies generate a framework for a model-based process control. Processes with nonlinear characteristics could benefit from advanced control schemes incorporating, for example, nonlinear controllers, multivariable control and optimization, or gain scheduling and narrow operating regions for the subset of linear controllers. The measurement and monitoring solutions for the SDR, however, need to be refined, and the advanced control could involve indirect measurements and observers as well.

### 3.3 RFB

A fluidized bed comprises an array of solid particles, which are suspended by an upward airflow. As the particles and bed grow, they are allowed to spill over a lip for collection and removal. New particles/nuclei are generated in the bed by attrition in the prevailing agitated environment. Rotating fluidized beds (RFBs) have high mixing performance and have been used in combustion applications for sewage sludge [51], coal [52], and wool scouring sludge [53]. Additionally, RFBs have been applied in wet granulation and coating applications [54–56], and polymerization [57,58]. Here, the emphasis is on RFBs in drying applications. Watano et al. [59] have studied the RFB in slurry drying. RFB variants, including pulsating elements, further

increase the application areas to homogenization, dispersion, extraction, adsorption, and absorption processes [60]. RFB reactors with static geometry have also been studied [61,62]. Other bed modification techniques have been reviewed in [63].

Considering the RFB in drying applications, rotational speed, solids/slurry feed flow rate, gas flow rate, and gas temperature can be manipulated, as indicated in Table 5. Along with the disturbances arising from the nature of the feed, they all interact in a complex manner with CVs, such as pressure drop, bed thickness, and gas fluidizing velocity. The constraints arise from the minimum fluidizing velocity (MFV), a gas flow, which will just ensure fluidization for a given rotational speed and gas temperature. Ideally, operation at speeds and gas flow rate just a little above the MFV is preferred. Increased gas flow leads to particle carryover and decreased gas flow to slumping (bed collapse). It is expected that the marker for the slumping phenomenon (at a given rotor speed) will be the pressure drop across the bed. However, the detailed behavior of the slumping phenomenon cannot be predicted by current theory. As the process is driven near its boundaries, optimal control schemes can be recommended. If the control range provided by the gas flow is not sufficient, multivariable control techniques are needed to take into account the interactions.

Numerical simulation of the RFB has been performed in several studies (see [54,57,58] and references therein). For coating and granulation applications, monitoring and control in a traditional fluidized bed have been recently reviewed in [64] and [65]. According to Burggraeve et al. [64], variations in the feed material should be minimized in fluid bed granulation, for example, by filtering, heating or cooling, and humidity removal of inlet air. It can be expected that these disturbances need to be considered also in RFB control because the bed depth is affected by the changes in the feed particle size and dryness, as well as from the difference between the solid feed rate and the particle discharge rate. The bed depth is an unknown function of the solid's flow rate, airflow (or air pressure drop), and rotating speed. The bed depth should be kept at its designed value.

### 3.4 TCR

The Taylor-Couette reactor (TCR; also referred to as Couette-Taylor or Taylor vortex) is an agitated cylindrical vessel in which the mixing is generated through a rotating inner cylinder positioned within a static outer cylinder. The movement of the inner cylinder and the opposing shear forces generate counter-rotating vortices in the annular gap through which the process material circulates. Very different flow regimes can be generated under different operating parameters, providing a wide range of mixing regimes that may be exploited for different products [66]. TCRs can be applied to different types of processes, such as photocatalysis, polymerization, precipitation/crystallization, and particle classification (see, e.g., [67–69]).

The primary MVs for the TCR are the rotational speed of the inner cylinder and the axial liquid flow rate. These govern the flow regime and dispersion/mixing, which can be related to application-dependent variables, such as particle properties (size, morphology), particle classification, and conversion. Temperature can be considered MVs or DVs affecting fluid viscosity and density, and, therefore, flow properties. Depending on the application, the system may also comprise gas flow rate control, reactant concentration control, and monitored variables such as pH [70]. The rotational speed and the agitation rate naturally affect the energy consumption of the process. The interaction table is depicted in Table 6.

As with many PI technologies with enhanced mass or heat transfer rates, the TCR also sets a challenging control problem with interacting variables and relatively fast dynamics. On the other hand, the reactor can provide a wide range of mixing regimes. Because the primary MVs (rotational speed, liquid flow rate) can be accurately adjusted, the TCR is well suited for a wide range of products, in cases where these MVs have an influence on critical product quality attributes and suitable models exist. At least for some potential applications, phenomenological models already exist in the literature. For example, hydrodynamics have been covered in [66,71] and the PSDs in [68,72]. On the other hand, studies covering continuous operation with process dynamics or control seem to be nonexistent. Therefore, it cannot be concluded if the TCR requires control strategies deviating from the ones developed for stirred tank reactors handling the same

materials and products. It can be expected that in the case of very short residence times, the typical PI challenges related to fast dynamics and advanced sensors need to be taken into account with TCR as well.

### 3.5 HP-SD

A heat pipe screw dryer (HP-SD) is a novel PI technology comprising an annular heat pipe and a screw conveyor. The annular heat pipe is a sealed, vacuum vessel containing a certain amount of liquid that evaporates and condenses along the length of the pipe to provide an indirect and passive heating system. The screw conveyor is used for continuous feed of wet material and displacement of dried material. HP-SD provides cost- and energy-efficient drying [73].

The operation of the HP-SD involves manipulating heat pipe temperature and screw speed with the latter variable affecting residence time. The primary CV is the product moisture content. In [73], the power consumption was also measured in order to calculate energy efficiency in terms of the specific moisture extraction rate. Additionally, the axial temperature profile of the heat pipe can be observed. The disturbances arise from ambient temperature and humidity, affecting the moisture extraction rate. The slurry feed flow rate can be considered a MV, or measured disturbance, depending on the application. In a process plant, the initial moisture content of the slurry may also vary and act as a disturbance. To calculate the energy efficiency, the initial moisture also needs to be measured. The interactions have been collected in Table 7.

The HP-SD forms a multivariable control problem. Without accounting for disturbances, there are two MVs (temperature, screw speed) and one CV (product moisture). Therefore, a model is needed to find optimized settings for the two MVs. If disturbances, feed flow rate, or energy efficiency are considered, the requirements for the monitoring solutions and control strategies change. Due to the expected interactions, model-based approaches are also recommendable in these cases. As the complexity of the process is relatively low, simple data-driven models are probably sufficient. On the other hand, the phenomena taking place are well known. Therefore, adjustment of existing mathematical models (see, e.g., [74]) for a

conventional screw dryer could also be straightforward. The performance of moisture control in an HP-SD could be improved by moisture prediction, allowing faster control actions. For example, in [75], control strategies with moisture prediction were developed for a rotary dryer.

### 3.6 HP-TSG

The heat pipe twin screw granulator (HP-TSG) is a twin screw extruder used for granulation processes with the addition of a heat pipe for potential performance improvement [76]. The principle of the heat pipe operation is similar to that described for the HP-SD. Seem et al. [77] have reviewed the available literature for twin screw granulation (TSG). Many of the findings presented in [77] are also valid for the HP-TSG.

The interaction table for the HP-TSG is presented in Table 8. Powder feed flow rate and liquid binder feed flow rate are the main MVs for a HP-TSG. These two typically work as a ratio control, where the liquid feed rate follows the powder feed rate. The screw speed is an additional MV, if needed. These all have an effect on granule size, the primary product quality attribute, as well as on granule porosity, flowability, and residence time distribution. Naturally, the applicable liquid levels are bounded to ensure that nucleation and granulation phenomena take place and avoid over wetting [77]. In a HP-TSG, product moisture also can be controlled by manipulating the jacket (heat pipe) temperature. The liquid flow rate or the liquid-to-solid ratio will also affect product moisture content. Feed particle size and ambient conditions (temperature and humidity) affect the granulation process as (observable) disturbances. Seem et al. [77] also noted that the barrel fill level and specific mechanical energy could be important factors in both comparison of different granulators and determination of operational costs. Motor torque indicates the degree of compaction and, hence, works as an indirect measurement during operation.

The development of advanced control strategies for TSG may require experimental testing as the current studies cannot comprehensively explain the granulation rate processes (nucleation, growth, breakage) taking place within TSG elements (see [77]). However, the PBM part of the multi-scale model in [78] could also be adopted to model-based control, as described, for example, in [22]. If fixed operational parameters

are preferred, and there are no strong disturbances, statistical process control (SPC) is one opportunity. Silva et al. [79] have developed a multivariate SPC strategy for a continuous tablet manufacturing line comprising a TSG. The monitoring and quality control system was able to detect the disturbances imposed, for example, in granulator barrel temperature, liquid mass flow, powder mass flow, and screw speed. However, [79] also observed that the process may return to a different steady state after perturbations, indicating irreversible process behavior and possibly requiring extra care in control design.

### 3.7 SFB

The swirling fluidized bed (SFB; or the toroidal fluidized bed, or the vortexing fluidized bed) involves the fluidization of solid particles in a swirling gas stream with improved mixing, reduced elutriation, and low pressure drops. SFB has the capability to process solids with a wide range of particle sizes. It is suited for applications with process retention times of less than a few minutes; with longer retention times, conventional fluidized beds and rotary kilns may be preferable [80]. Process applications consist of, for example, combustion of biomass and poultry waste [81,82], combustion of biomass [83], and drying [84].

The operation of a SFB can be affected by manipulating the solids feed rate, gas feed rate, bed temperature, and swirling intensity. The air-fuel ratio can be considered instead of separate flow rates for the solids and gas. Indeed, the feed control to the small bed of the reactor has been recognized as a critical issue [80]. Temperature control may involve annular cooling coils [85], water injected to the bed [86], or a preheater for the inlet gas temperature. The swirling intensity is determined by secondary gas flows. The operation may be disturbed by the gas temperature changes, feed solids moisture content, and PSD. Naturally, PSD could be controlled using a filtering step, and the feed moisture content could be regulated using a combination of pre-wetters/dryers at the expense of power demand.

In Table 9, eight potential CVs for the SFB are presented: particle mass flux, bed height, bed temperature distribution, solids velocity distribution, gas composition, gas pressure drop, product humidity/dryness, and throughput. In Chyang et al. [86], a dynamic stability constant was proposed to describe the inertia characteristics of the vortexing fluidized bed combustor. The stability constant is indicative of the speed of



response and depends on the operating conditions. The bed pressure drop can be monitored because it can be seen to indicate the fouling of blades and other surfaces that affect process performance.

A number of studies have been done regarding the numerical simulation of SFB. For example, Ridluan et al. [87] have tested four different turbulence models within the numerical study of the swirling/recirculation flow in a 3D vortex combustor. Experimental studies for predicting the combustion efficiency for different fuels and operational parameters and the effect of operational parameters to nitrogen emission have also been given in [82] and [88], respectively. There seem to be no journal articles dedicated to process control of SFB. However, the control studies for the close counterpart technologies (RFB, conventional fluidized bed, and rotary kiln) may give some insight.

### 3.8 Summary

The process control considerations for a range of selected PI technologies directed to solids handling processes have been discussed. The findings are summarized in Table 10, where the number of identified variables is given along with important references. It is obvious that not all the variables related to the potential PI applications could be treated, especially in terms of strength and speed of interaction. Such information, along with the determination of a final set of CVs and control objectives, require application-dependent process knowledge. In addition, the information collection exercise showed that it is somewhat easier for the PI experts to relate the variables to mechanical or hydrodynamic performance of the PI equipment than to the wide range of potential process applications. With an additional insight into the available phenomenological models, a process development team interested in a particular PI technology can, however, connect the hydrodynamic variables to application-dependent process variables and make use of the qualitative control information. As shown in Table 10, such models exist for most of the studied PI technologies. On the other hand, studies focusing particularly on control problems related to these technologies are almost nonexistent. It will be important to report the operational experiences of PI applications in solids handling processes in order to complement the data generated in this study and to perform a deeper analysis with selected PI technologies and applications. With positive feedback and

experiences reported, one obstacle between a PI technology and its successful implementation would be diminished.

The data presented in this contribution could benefit from detailed considerations about monitoring solutions for each PI technology. For example, during the information collection, it was recognized that SDR operation would benefit from miniaturized sensors for standard process measurements (e.g., temperature, film thickness) and noninvasive techniques for reaction monitoring. More importantly, instrument response times need to match the fast dynamics in SDR to enable real-time control. These challenges are typical for any kind of PI, and the distributed nature of the particulate material adds another dimension to the problem. In the field of pharmaceutical tablet manufacturing, the process analytical techniques (PAT) used for process monitoring and control have been reviewed in [89], and a more extensive summary of PAT is given in [23].

The next steps in the process control design require application-dependent, quantitative information in the form of experimental data or mathematical models. One simple procedure supporting the preliminary design stage with steady-state information is described in [33]. The process dynamics (dead times, time constants, instrument response times) play a crucial role in the detailed design stage. Maya-Yescas et al. [90] have recently treated this topic with respect to PI in chemical processes. While the preceding considerations are mostly targeted to new process designs, PI implementation may be directed to existing processes as well. In this case, the interest lies also in the required changes in process operation. If PI is to change the process dynamics and monitoring solutions considerably, the plant-wide control aspects need to be accounted for as part of the PI project.

## 4 Conclusions

Process intensification, whether it considers a new process design or a redesign with new equipment, involves a risk that the estimated efficiency cannot be reached during operation. The reasons for this are often related to the difficulties in controlling the plant at its designed operation points. Especially with

solids handling processes, the distributed nature of the process and complex dynamics increases these risks. To avoid such bottlenecks, the control issues need to be considered in an integrated manner with the process design. Hence, the evaluation of the requirements for process control can be considered as one key requirement for applying PI to a given solid handling process. In this article, such information was generated for selected PI technologies. The information provided should diminish the gap between PI and control and help the process development team reach a successful PI implementation.

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## Appendix

The focus of this control questionnaire is to identify the available manipulated variables in your PI technology for the use of automated (advanced) process control. Correspondingly, the possible controlled variables, disturbance variables, and other observable variables that may be relevant in different PI applications are identified. The systematic procedure modified from (Roffel & Betlem 2006)<sup>2</sup> is considered here, with some additional questions. Any input from the PI experts is welcome. If the systematic procedure leading to an interaction table cannot be defined, please move directly on to the additional questions presented.

Checklist for the systematic procedure:

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<sup>2</sup> Process dynamics and control: modeling for control and prediction, B. Roffel & B. Betlem, 543p., John Wiley & Sons Ltd., 2006

- 1) Describe the process (explain the PI equipment working principle and example process applications, and provide any relevant material or references).
- 2) Define the goals of operation in a selected application(s); quantify if possible.
- 3) Investigate process boundaries and external disturbances.
  - a. Define the typical positioning of the investigated PI in the process chain/plant.
  - b. Define any auxiliary processes, such as steam, electricity, flue gas, or exchange of material.
  - c. Evaluate the expected disturbances and define whether they are measurable. This should cover both the internal disturbances (due to kinetics, flows, fouling, etc.) and the external disturbances (due to environment, other subprocesses, etc.).
- 4) Define potential controlled variables (controlled qualities, important process design parameters).
- 5) Define manipulated (correcting/adjustable) variables (controlled process conditions, controlled material contents).
- 6) Arrange the controlled variables (columns) and manipulated variables (rows) into an interaction table (as in the example provided).
- 7) Establish the power and speed of the control in the interaction table using qualitative measures such as large, small, moderate, and nil (for power/magnitude), and slow, fast, fair, and nil (for speed/dynamic response). The scale is dependent on the fastest/largest response.

Additional questions:

- If the interaction table could not be defined, list the possible manipulated variables, controlled variables, and measured and unmeasured disturbances.
- Specify any existing measurements and controls, or recommendations, for the investigated PI based on test rigs, industrial implementations, or literature sources you are aware of.
- Specify any existing models of the investigated PI.

- Provide any experimental results (e.g., design of experiments, other experiments, scientific papers, technical reports, etc.) if possible.
- If automated control is not feasible, what variables could be monitored, for example, for use with the statistical process control (SPC)? Additionally, in this case, define the possible manipulated variables, as well as measured and unmeasured disturbances.

ACCEPTED MANUSCRIPT

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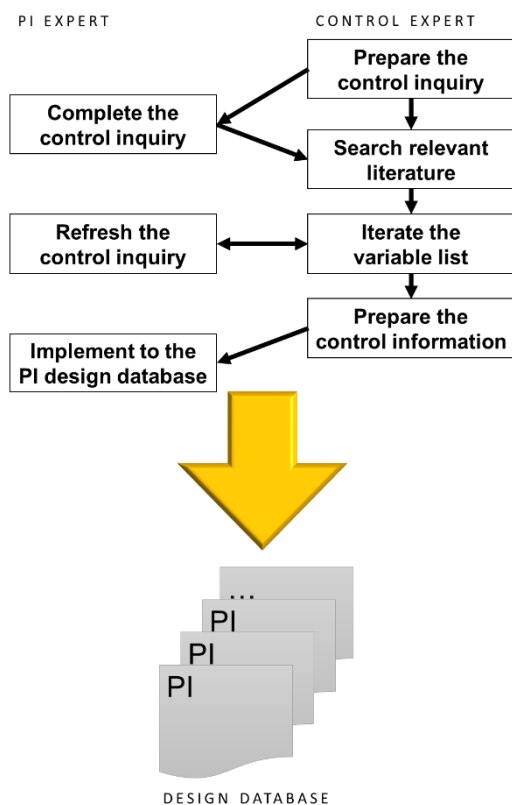


Figure 1. Control information collection workflow.

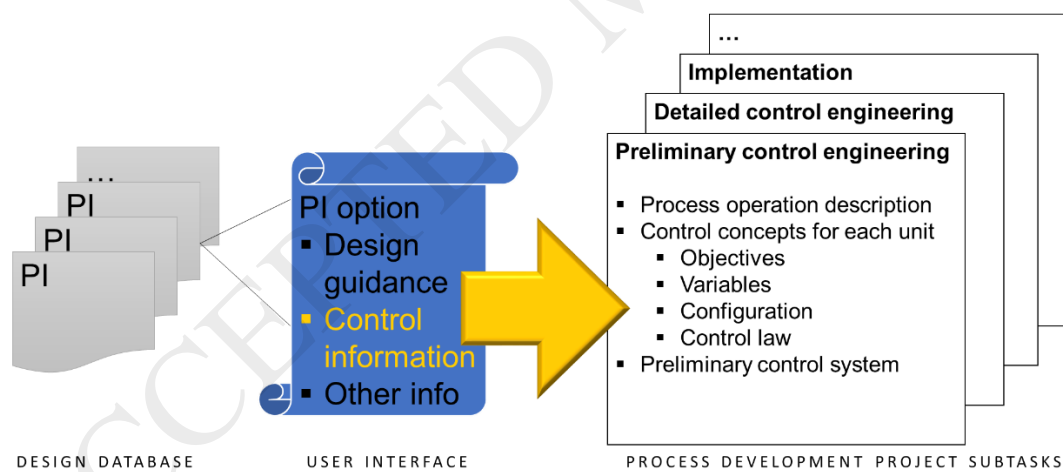


Figure 2. Control information as a part of PI implementation.



**Table 1.** Studied PI technologies and their applications.

<b>PI technology</b>	<b>Acronym</b>	<b>Applications</b>
<b>Oscillatory baffled reactor</b>	OBR	Precipitation/crystallization, Catalytic reactions
<b>Spinning disc reactor</b>	SDR	Precipitation/crystallization, Catalytic reactions, Bioprocessing
<b>Rotating fluidized bed</b>	RFB	Particle coating, Drying, Thermal processing
<b>Taylor-Couette reactor</b>	TCR	Precipitation/crystallization, Catalytic reactions, Granulation, Mixing
<b>Heat pipe screw dryer</b>	HP-SD	Drying
<b>Heat pipe twin screw granulator</b>	HP-TSG	Granulation/drying
<b>Swirling fluidized bed</b>	SFB	Separation, Drying, Thermal processing

**Table 2.** Fictitious interaction table. The input (manipulated and disturbance) variables can be found from the columns, and the output (controlled and observed) variables from the rows.

		MV			DV	
		Mixing speed	Feed flowrate	Reactor temperature	Feed moisture	Feed particle density
CV	Flow regime	Large Fast	Moderate Fast	nil	nil	Small unknown
	Product particle size	Moderate Fair	Moderate Slow	nil	nil	Moderate Slow
	Product moisture	Small Slow	Small Slow	Moderate Fair	Large Slow	Small Slow
OV	Power consumption	Small Fast	nil	Large Fast	nil	Moderate Fair

**Table 3. Interaction table for the oscillatory baffled reactor.**

OBR		Input variables (MV, DV)				
		Feed rate	flow	Oscillation frequency	Oscillation amplitude	Temperature profiles
Output variables (CV, OV)	Residence time	Large Fast		Nil	Nil	Nil
	Solid suspension behavior	Nil		Large Fast	Large Fast	Nil
	Plug flow behavior	Nil		Large Moderate	Large Moderate	Nil
	Temperature	Large Fast		Large Fast	Large Fast	Large Fast
	Product PSD (polymer)	Nil		Large Moderate	Moderate Moderate	Nil
	Conversion	Large Fast		Nil	Nil	Nil
	Yield	Moderate Fast		Large Fast	Large Fast	Nil
	Selectivity	Moderate Moderate		Nil	Nil	Nil
	Reactor pressure	Nil		Nil	Nil	Nil
	Throughput	Large Moderate		Nil	Nil	Nil

**Table 4. Interaction table for the spinning disc reactor. The speed/dynamic responses are defined as fast – seconds, fair – minutes, slow – hours.**

SDR		Input variables (MV, DV)		
		Rotational speed	Total feed flowrate	Temperature
Output variables (CV, OV)	Residence time	Large Fast	Large Fast	Small-moderate Fair- Fast <sup>1</sup>
	Film thickness	Large Fast	Large Fast	Small-moderate Fair- Fast <sup>1</sup>
	Shear rate (Mixing)	Large Fast	Moderate Fast	Small-moderate Fair- Fast <sup>1</sup>
	Particle size and distribution	Large Fast	Moderate-Large Fast	Small-moderate Fair- Fast <sup>1</sup>
	Conversion	Large <sup>3</sup> Fast	Moderate-Large <sup>3</sup> Fast	Moderate-Large <sup>2</sup> Fair
	Yield	Large <sup>3</sup> Fast	Moderate-Large <sup>3</sup> Fast	Moderate- Large <sup>2</sup> Fair
	pH	Large <sup>3</sup> Fast	Moderate-Large <sup>3</sup> Fast	Moderate- Large <sup>2</sup> Fair

<sup>1</sup> Temperature affects fluid properties (density and viscosity primarily), which impact design output variables (residence time, film thickness, and shear rate).

<sup>2</sup> Depends on rate equations, but generally a 10°C rise in temperature doubles the rate for most reactions; dynamic response is expected to be fair due to a new steady state having to be attained.

<sup>3</sup> Conversion, yield, and pH are all directly dependent on a combination of design output variables (residence time, film thickness, and shear rate); hence, their variation with the design input variables (rotational speed and feed flow rate) is similar to those between the design input and design output variables.

**Table 5. Interaction table for the rotating fluidized bed.**

RFB		Input variables (MV, DV)					
		Rotational speed	Solids/slurry feed flow rate	Gas feed flow rate	Gas temperature	Feed moisture content	Gas humidity
Output variables (CV, OV)	Pressure drop	Large Fast	Large Fast <sup>5</sup>	Large Fast <sup>3</sup>	Small Small	Small Small <sup>6</sup>	Small Small
	Gas fluidizing velocity	Large Fast	Large Fast	Large Fast <sup>2</sup>	Nil	Nil <sup>5</sup>	Small Small
	Bed thickness /depth/loading	Large Fast <sup>4</sup>	Large Fast <sup>4</sup>	Large Fast <sup>4</sup>	Nil	Small Small <sup>5</sup>	Small Small
	Residence time	Large Fast <sup>1</sup>	Large Slow	Modest Slow	Large Fast	Large Small	Large Small
	Exhaust air temperature	Small Small	Nil	Nil	Large Fast	Modest Slow	Modest Slow
	Bed temperature	Nil			Large Fast	Modest Fast	
	Product throughput	Large Fast	Large Fast		Nil		
	Product PSD	Large Fast		Nil	Nil	Small Slow	Small Slow
	Product temperature	Nil	Nil	Modest Small	Large Fast	Modest Fast	Small
	Product moisture content	Small Small			Small Fast	Small Fast	Large Slow
	Flue gas composition	Nil	Large Slow	Large Slow	Nil <sup>7</sup>	Modest Slow	Modest Slow

<sup>1</sup> A strong function of the product being treated.

<sup>2</sup> Controlled after one reaches the minimum fluidizing velocity.

<sup>3</sup> More a function of the bed's outer radius and the critical fluidizing velocity based on this.

<sup>4</sup> The depth of the bed needs to be controlled—it is a function of feed flow rate but also rotating speed and fluidizing gas velocity.

<sup>5</sup> The nature of the feed can vary significantly—a slurry will give markedly different results than a particle feed of particles with relatively low moisture content. Experiments may be needed to obtain optimum conditions.

<sup>6</sup> A function of the moisture content. Modest for most drying applications, but a slurry would have a greater influence. Again, experiments would be needed.

<sup>7</sup> More a function of the product type/reaction.

**Table 6. Interaction table for the Taylor-Couette reactor. The speed/dynamic responses are defined as fast – seconds, fair – minutes, slow – hours.**

TCR		Input variables (MV, DV)		
		Rotational speed	Axial liquid flowrate	Temperature <sup>1</sup>
Output variables (CV, OV)	Flow regime (measured by ratio of rotational Re to critical Re)	Large Fast	Large Fast	Moderate Fair- Fast
	Dispersion	Large Fast	Large Fast	Moderate Fair- Fast
	Particle size and distribution	Large Fast	Moderate Fast	Moderate Fair- Fast
	Particle classification	Large Fair	Moderate Fast	Moderate Fair- Fast

<sup>1</sup> Temperature affects density and viscosity of working fluid, which, in turn, affect the controlled parameters.

**Table 7. Interaction table for the heat pipe screw dryer. The speed/dynamic responses are defined as fast – seconds, fair – minutes, slow – hours.**

HP-SD		Input variables (MV, DV)			
		Slurry feed rate	Initial moisture content	Screw speed	Heater band temperature
Output variables (CV, OV)	Final moisture content	Small Nil	Small Nil	Moderate Fair	Large Fair
	Energy efficiency <sup>1</sup>	Small Nil	Small Nil	Moderate Fair	Large Fair
	Temperature differential of the heat pipe (axial)	Small Nil	Small Nil	Moderate Nil	Large Slow

<sup>1</sup>Measured in terms of specific moisture extraction rate (kg-water/kWh).

**Table 8. Interaction table for the heat pipe twin screw granulator. The speed/dynamic responses are defined as fast – seconds, fair – minutes, slow – hours.**

HP-TSG		Input variables (MV, DV)			
		Powder feed rate or barrel fill level	Liquid-to-solid ratio or liquid flow rate	Screw speed	Jacket or heat pipe temperature
Output variables (CV, OV)	Granule size distribution (PSD)	Moderate Fair	Large Fair	Moderate Fair	Nil
	Granule porosity (related to density)	Large Nil	Large Nil	Small Nil	Nil
	Granule flowability	Large Nil	Large Nil	Small Nil	Nil
	Granule moisture content	Nil	Moderate Fair	Nil	Large
	Residence time distribution (RTD)	Large Fast	Moderate Fair	Large Fast	Nil



**Table 9. Interaction table for the swirling fluidized bed.**

SFB		Input variables (MV, DV)						
		Solids feed flow <sup>1</sup>	Inlet gas flow <sup>1</sup>	Swirling intensity <sup>2</sup>	Bed temperature <sup>3</sup>	Gas temperature <sup>3</sup>	Feed solids moisture <sup>4</sup>	Feed solids PSD <sup>4</sup>
Output variables (CV, OV)	Bed temperature (distribution)	Small Slow	Moderate Slow	Moderate Fast	Large Fast	Large Fast	Nil Slow	Nil Slow
	Solids velocity (distribution)	Nil	Moderate Fast	Moderate Fast	Nil	Nil	Nil	Nil Fair
	Bed height	Small Fast	Moderate Fast	Moderate Fair	Nil	Nil	Nil	Nil Slow
	Particle mass flux	Small Fair	Moderate Fast	Nil	Nil	Nil	Nil	Nil Slow
	Gas pressure drop	Small Fast	Moderate Fast	Moderate Fast	Nil	Large Slow	Nil Slow	Nil Slow
	Gas composition	Nil	Moderate Fast	Nil	Nil	Large Fast	Nil Slow	Nil
	Product humidity (dryness)	Small Fair	Moderate Slow	Moderate Fair	Large Fair	Large Fair	Nil Fast	Nil Slow
	Throughput	Small Fair	Moderate Fast	Nil	Nil	Nil	Nil	Nil Slow

<sup>1</sup>Air-fuel ratio can be considered instead of separate flow rates.

<sup>2</sup>Swirling intensity is determined by secondary gas flows.

<sup>3</sup>Bed temperature is manipulated with the cooling coils around the reactor and/or the preheater for the inlet gas temperature.

<sup>4</sup>Assumed as uncontrollable disturbances. If necessary, the PSD could be controlled using a small amount of power (e.g., by adding in a filtering step), while the feed moisture content could be regulated using a combination of pre-wetters/dryers (moderate to high power demand).

**Table 10.** Synthesis from the information collected for the studied PI technologies.

	<b>OBR</b>	<b>SDR</b>	<b>RFB</b>	<b>TCR</b>	<b>HP-SD</b>	<b>HP-TSG</b>	<b>SFB</b>
<b>Number of inputs defined</b>	4	3	6	3	4	4	7
<b>Number of outputs defined</b>	10	7	11	4	3	5	8
<b>References to earlier control studies</b>	None	[18,19]	None	None	None	None	None
<b>References to support model-based control design</b>	[41] [38] [42,43]	[44] [46]	[50] [54,57,58 ]	[66,71] [68,72]	None	None	[87]
<b>References from analogous techniques to support control design</b>	None	None	[64] [65]	None	[74] [75]	[77] [78] [79]	[64] [65]