SPIRE 8 – 2015 – Solids handling for intensified process technology	

Title: Intensified by Design© for the intensification of processes involving solids handling

Acronym: IbD

Grant Agreement No: 680565

Deliverable 4.3	Publishable / Public outputs from WP4
Associated WP	WP4: PAT and Control solutions of intensified processes
Associated Tasks	All tasks
Due Date	31/08/2017
Date Delivered	10/09/2018
Prepared by (Lead Partner)	VTT
Partners involved	OULU, IRIS, Tel-Tek, ZHAW, FREEMAN
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Dissemination Level	Public (PU)

Table of Contents

1. Introduction

WP4 of the IbD project focused on process analytical technologies (PAT) and control solutions of intensified processes.

The main partners involved with WP4 are IRIS, VTT, ZHAW, OULU, Tel-Tek and Freeman.

2. Objectives of WP4

The original objectives for work package 4 can be summarized as follows:

- Define novel sensors and process analytical devices, advanced statistical and chemometric methodologies, and advanced process control for the intensification of processes involving solids handling.
- Provide tools for identification of intensified processes.
- Provide methods for utilizing indirect measurements in control.
- Provide control strategies for intensified processes.

However, there are some additional objectives for WP4. These stem from the fact that the main outputs of the IbD project are the IbD platform (WP5) and successful case studies (WP6). Thus, the additional objectives of WP4 support other work packages:

- Provide the PAT and Control Toolbox for the IbD platform (supports WP5).
- Provide PAT and control solutions for the case studies (supports WP6).

In addition, WP4 has closely cooperated with WP3.

3. Key achievements of WP4

- A state of the art review in analytical technologies and metering solutions suitable for PI control involving solids handling was compiled as the first task of WP4. The work included a review of process analytical tools (PAT) suitable for powder processing, the basic principles of chemometrics, probes and their interfaces, as well as the cleaning of probes, measurement heads and windows. The basic principles and applications of the theory of sampling was reviewed, as well as metering solutions, which covers both measuring powder flow properties and in-line measurement of powder flow rate.
- The WP4 team has conducted proof-of-principle tests of many PAT methods that have applications in the IbD project case studies. Raman spectroscopy has been demonstrated in pharmaceutical and mineral processing applications, miniaturized NIR in pharmaceutical applications, and camera-based particle characterization methods in mineral processing applications. Tel-Tek has demonstrated the potential of using acoustic sensors in online process analysis of powder mixtures.
- The FT4 powder rheometer was used to conduct granule characterisation on samples of pharmaceutical final blends (case study 4.2) to investigate relationships with Critical Quality Attributes (CQAs) of the resulting tablets. A relationship between tablet hardness with a combination of dynamic flow, bulk and shear properties was demonstrated.
- FT4 analysis was also conducted to study the differences between pharmaceutical blends that were dried using intensified vs standard drying equipment. Differences in the rheological

properties were observed, as a direct consequence of the modified and intensified process, and also the milling operation.

- ZHAW developed and improved measurements of powder wettability with different liquids from which the contact angle can be measured. ZHAW compiled a database of surface energies for powders measured with Washburn method (own measurements and literature data). In addition, three recommendations for using the surface energy database in aiding the design of PI processes were proposed.
- An FT4 Powder Rheometer was used to characterize nanoparticles with comparable particle size distributions but different chemical compositions. The relationships between surface properties and bulk rheological properties were investigated.
- The process control point-of-view on modelling, measurements, and control aspects of continuous solids handling processes was studied in a state-of-the-art survey. The literature search was aimed for solids handling operations in mineral beneficiation, pharmaceutical processes, and crystallization.
- Process identification methods were casted into a flowchart in order to provide a modeling advisor functionality to the IbD® platform. The modeling advisor allows to screen and select the modeling methods needed for process control or soft sensor development.
- The PAT and Control Toolbox for the IbD platform was developed based on the research in WP4. The Toolbox includes tools for initial process analysis, PAT advisor and the Control advisor. These were implemented in the IbD platform as a part of WP5 activities. The initial process analysis step guides the user through control questionnaire, variable listing, sensitivity analysis and interaction table. This step results in a list of potential controlled and manipulated variables and expected interactions and disturbances. Once the variables are known, the user can proceed to the PAT advisor. The PAT advisor asks the user about PAT needs, and then shows scoring of different PAT instrument types based on the user's answers, as well as some alternative instrument suppliers. In addition, general guidance on acquiring and installing the PAT instrumentation is given. The control advisor step includes arranging the previously listed variables into an interaction table, identifying possible variable pairing for the univariate control loops and analysing the controllability of the system. In order to perform the variable pairing and controllability analysis, quantitative data on the interactions need to be available.

4. Public outputs from WP4

4.1. Journal papers

Published papers

M. Ohenoja, K. Boodhoo, D. Reay, M. Paavola, K. Leiviskä, "Process control in intensified continuous solids handling", Chemical Engineering and Processing - Process Intensification 131, pp. 59 - 69, 2018.

D. Wu, B. P. Binks, A. Honciuc, "Modeling the Interfacial Energy of Surfactant-Free Amphiphilic Janus Nanoparticles from Phase Inversion in Pickering Emulsions", Langmuir 34 (3), pp. 1225-1233, 2017.

M. Ohenoja, M. Paavola, K. Leiviskä, (accepted), "Control design tools for intensified solids handling process concepts". In: Advances in Systematic Creativity - Creating and Managing Innovations. Eds: Chechurin, L., Collan, M. Palgrave Macmillan, 2019. doi: 10.1007/978-3-319-78075-7

Papers under review or in preparation

S. Uusitalo, T. Soudunsaari, J. Sumen, K. Rahkamaa-Tolonen, O. Haavisto, J. Kaartinen, J. Huuskonen, J. Paaso, "On-line analysis of minerals from sulphide ore using NIR Raman spectroscopy", In preparation (December 2018).

4.2. Conference publications

Conference presentations

M. Ohenoja, M. Paavola, "Control design tools for intensified solids handling processes". TRIZ Future Conference 2017, The 17th International Conference of the European TRIZ Association, Lappeenranta, Finland.

Conference posters

M. Ohenoja, 2017, "Control perspective of selected PI technologies in continuous solids handling", 10th World Congress of Chemical Engineering – International Process Intensification Conference (WCCE-IPIC), 01-05 October 2017, Barcelona, Spain. https://doi.org/10.13140/RG.2.2.27014.47687.

R. Sarjonen, E. Hietala, J. Tenhunen, J. Paaso, "Raman measurements in mineral processing", Optics & Photonics Days 2017, May 2017, Oulu, Finland.

4.3. Other technical presentations (e.g seminars, invited talks etc.)

M. Edelmann, A. Hämmerli, C.A. Ziegler-Meyer, C. Adlhart "Quality assurance of packed tablets by contact free near-infrared spectroscopy" Factories of the Future: ICBT Day of Life Sciences, Switzerland, June 22nd, 2017.

M. de Lourdes Aja Montes, J. Krauser, D. Lührs, C. Adlhart "Increasing levels of detections by vibrational and functional group labelling" Swiss Raman Users Meeting, May 12th, 2016.

4.4. Non-technical presentations

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5. Appendices

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