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 6.5	Publishable PI Case Studies
Associated WP	WP6 PI Case Studies Implementation
Associated Tasks	Task 6.1 Standardised Approach to the Implementation of the PI Case Studies Task 6.2 Design and Planning of the Demonstrators. Task 6.3 Building and Testing of Demonstrators Task 6.4 Validation of the Demonstrators. Task 6.5 Analysis and Recommendations for Further Scale-Up.
Due Date	31/05/2018
Date Delivered	14/09/2018
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Partners involved	All
Authors	David Reay (DRA) and the individual case study teams
Dissemination Level	Public (PU)

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1. Introduction

This report details the publishable outcomes of IbD Work Package 6: PI Case Study Implementation. In this work package, 6 case study teams (made up of host industrial plants, academics, modelling experts, PI experts, process analytical technology (PAT) experts, fouling experts and life cycle analysts) designed, modelled, built, demonstrated and validated intensified technologies according to the following tasks:

Task 6.1 Standardised Approach to the Implementation of the PI Case Studies. Each case study utilised standardised tools which form the basis of the IbD platform to guide their approach to implementing PI technologies into their processes. This included the KBE system (tool for shortlisting intensified technologies), the TRIZ methodology (tool for novel idea generation/problem solving), various built-in-modules (where applicable) to aid design tasks and the fouling/PAT toolbox to aid ancillary system design.

Task 6.2 Design and Planning of the Demonstrators. Each case study designed a demonstrator unit for implementation on their process (or a model/pilot process in some cases). A comprehensive experimental programme was also devised for each case study to evaluate the benefits of the PI technology (from the point of view of cost savings, energy savings, life cycle benefits, *etc*.)

Task 6.3 Building and Testing of Demonstrators. Each case study built and commissioned a demonstrator unit for validation, including PAT systems and consideration for upstream/downstream processing.

Task 6.4 Validation of the Demonstrators. Each case study performed comprehensive experiments to determine the benefits of the PI technology, and to compare the results with modelling results from the design phase.

Task 6.5 Analysis and Recommendations for Further Scale-Up. Each case study performed a comprehensive analysis of data from the previous tasks in order to make final conclusions and recommendations for the future implementation of the PI technology they investigated. Depending on the case study, this included recommendations for scale-up of the technology to production scale (although some case studies operated at production throughput throughout the work package), recommendations for upstream/downstream modifications required for uptake of the technology and an evaluation of the technology's current TRL (technology readiness level).

This report details the public (i.e. not confidential) outputs from each of these tasks and is organised into subsections (Sections 2-7) by each case study. In Section 8, a reference list of other public outputs from this work package is presented (journal papers, conference presentations, miscellaneous). Table 1 overleaf summarises the technologies investigated in each case study and their TRL levels (for the specific processes investigated) following the IbD project.

Case Study	Host Company	Brief Description	PI Technology	TRL following WP6 Tasks
CS1: Metallic Powder Processing	MBN Nanomaterialia spa, Treviso, Italy	Intensification of classification of metallic powders	Elbow Jet Classifier	7
CS2: Ceramic Powder Processing	Euroatomizado, Castellon, Spain	Intensification of dry granulation process as part of a wider drive to switch from a wet process route to a dry process route	High Shear Mixer Granulator	7-8
CS3: Mineral Beneficiation	Outotec Oyj, Espoo, Finland (Equipment supplier) Pyhasalmi Mine Oy, Pyhäsalmi, Finland (End user)	The mineral beneficiation process chain intensification seeks for energy and raw material savings of grinding and flotation via coarse flotation and variety of PAT tools.	Coarse Flotation Device Model Predictive Control Raman Spectrometer, Machine Vision, Electrical Resistance Tomography Dynamic Modelling and Adaptation	5-8
CS4.1: Pharmaceutical Processing I	Sanofi-Aventis, Barcelona, Spain	Intensification of granulation process	Twin Screw Granulator	6
CS4.2: Pharmaceutical Processing II	Industrias Farmacéuticas Almirall, S.A. Sant Andreu de la Barca, Spain	Intensification of drying process	Spiral Flash Dryer	6-7

CS5: CS5: Intensification of Chemical Processing Involving Solid Reagents	AM Technology, Runcorn, UK.	Intensification of multiphase chemical reaction technology using an advanced mechanically agitated flow reactor	CoFlore Reactor	5

2. CS1: Metallic Powder Processing

Case study host:

MBN Nanomaterialia spa – Treviso - Italy.

Case study leader:

Company, City, Country.

Case study team:

List of: Company, City, Country.

Brief description of process unit(s) of interest for intensification and motivation:

The case study aims at solving the classification task of the high-density particles produced by ball milling at MBN Nanomateralia. The particles are used in the production of wear resistant thick coatings.

The material must be sprayed by thermal spraying and for this reason requires to be selected in the narrow particle size range of 10 – 38 μm. Hence, **the main target of the PI for MBN is to put in the production line the different classification steps after the ball milling with a recirculation of the coarser fraction.** This, per se, will constitute a big advancement in the intensification of the process. To do this, some powder classification technologies should be changed (i.e. sieving) and in-line monitoring of the overall process should also be implemented to manage the process.

Currently MBN is classifying the particles in separated steps, with standard sieving and classifying equipment available for powder metallurgy sector that have limits regarding the processing due to the wear and the safety issue of the specific material used. **The aim is to move to a continuous or semi-continuous process to decrease the process costs and increase process yields.** Moreover, **limiting handling operation will decrease the risk of exposure to cobalt (Co) which is carcinogenic, for instance, sieving is a simple and easy process but requires substitution of the meshes that are often broken by this hard material. Due to the risk of exposure to Co, this maintenance work is not easy and would be limited as much as possible.**

Brief description of PI technology chosen:

The chosen PI technology is the Elbow Air-Jet Classifier (hereafter, EJAC). It can classify particles of different size and has the advantages of not using water, magnetic fields, heating, etc. Hence, it is a quite simple element that just needs air streams. The main design difficulty is its shape (curvature to enter in the different channels and lengths between them).

Currently this classification system is available in the market from a few producer outside Europe, and applied by toner producers to separate the smallest particles.

The EJAC working principle has been revised and applied to the separation of the broad particle size distribution in four different ranges: (1) the finest particles, (2) the product for Thermal Spraying, (3) medium sized particles used in other product and (4) bigger particles to be fed back to the milling plant.

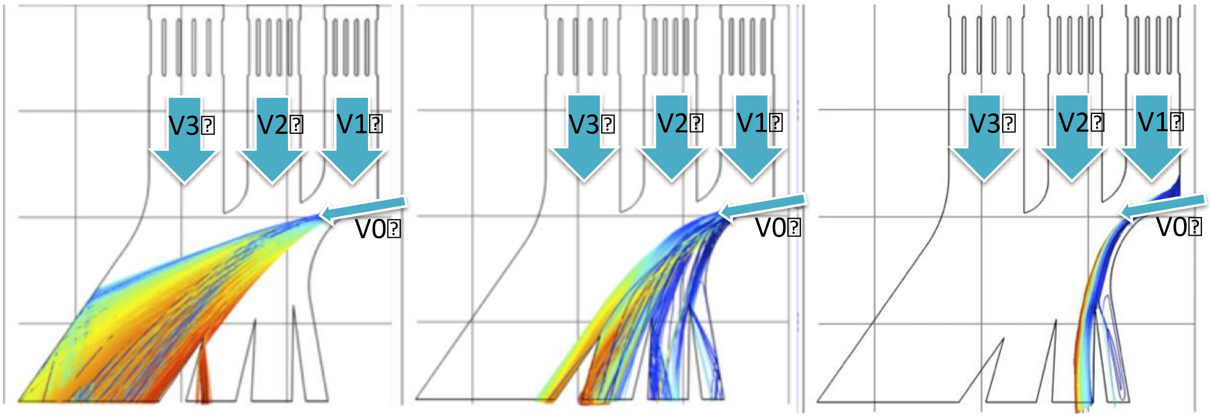


Figure 1 Simulation of particle trajectories in the EJAC, the optimal solution is obtained acting on inlet air velocities (V0 to V3)

Brief summary of results:

The EJAC unit has been realized from scratch, after a complete modelling of the powder dynamic in the classification chamber in response to the velocities V0 to V3 (Figure 1) and in different geometrical configurations.

The validated design has been realized in transparent plastic to evidence particle flows and points of most wear. Three air blower has been installed to provide the necessary air to curve the particle trajectories in the four outlet.

Two cyclones has been installed for the medium fraction and the product fraction, collecting drums with filters has been chosen for the coarse fraction and the smaller fraction.

The operation are controlled by an external PLC that drives the blowers and collect the data form the installed sensors that measure air flow, pressure, material flow and collected product amount.

Figure 2. From the left: maon chamber with analogic pressure control, the installed blowers, the cabin for test confinement

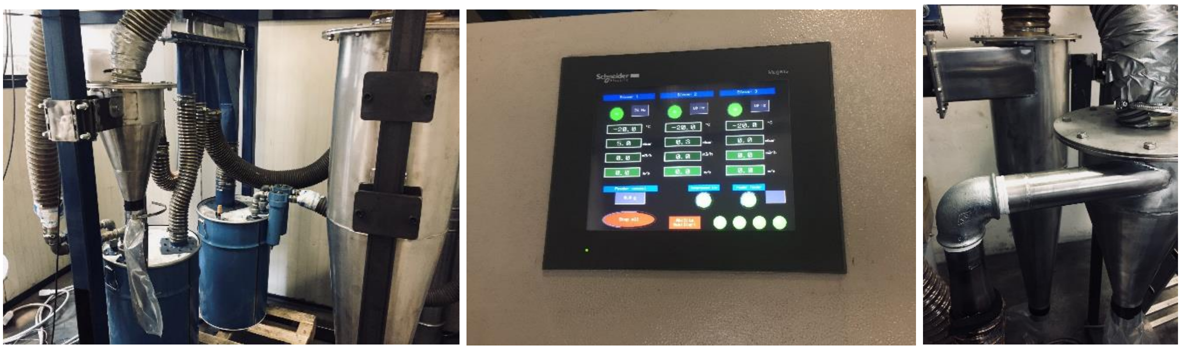
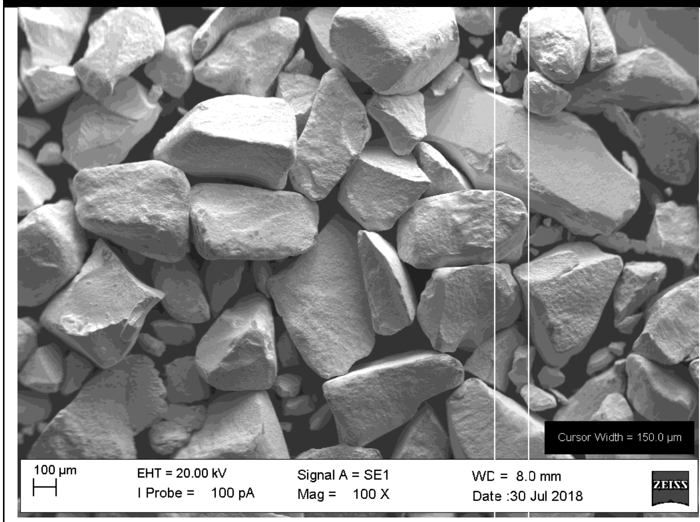
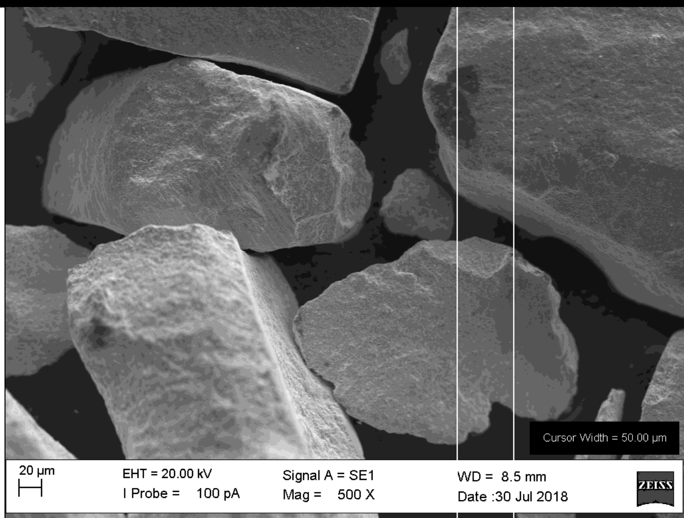
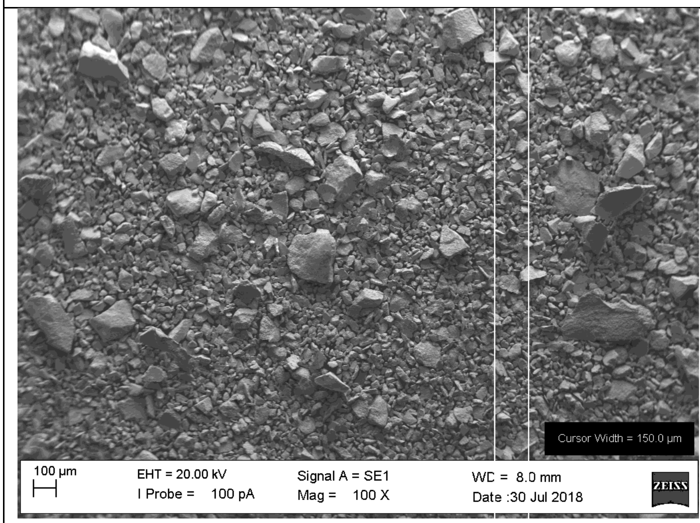


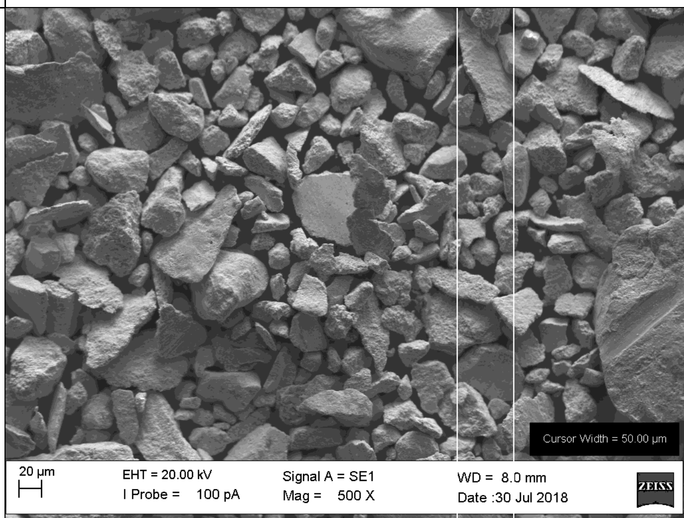
Figure 3 From the left: the cyclones and collecting drums, the PLC running the project, detail of the installed cyclones

The EJAC principle works with the heavy Ceramic-Metal composites tested, the images below are examples of the particles obtained as a results of the classification.

<table>
<tr><th>Magnification 100X</th><th>Magnification 500X</th></tr>
<tr><td>

Coarse Fraction – 4th outlet</td><td>

Coarse Fraction – 4th outlet</td></tr>
<tr><td>

Medium Fraction –3rd outlet</td><td>

Medium Fraction –3rd outlet</td></tr>
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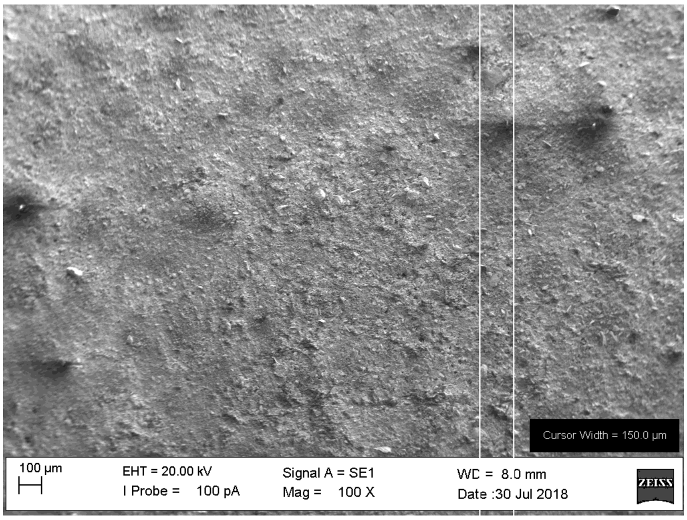
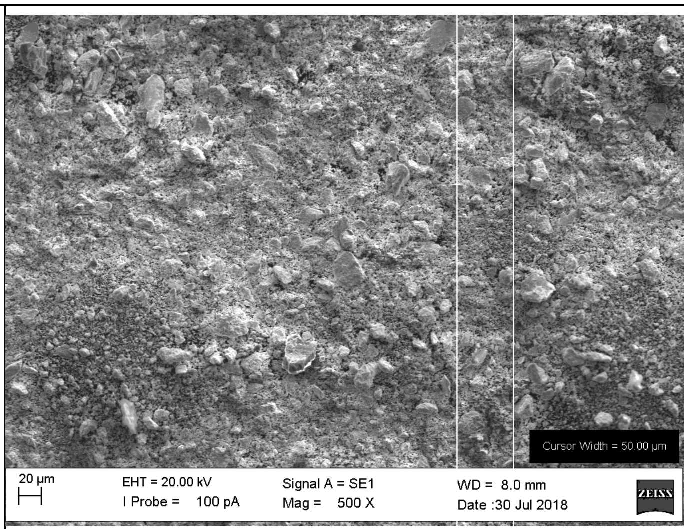


Product Fraction –2nd outlet



Product Fraction –2nd outlet

Final conclusions from case-study:

The PI is a success and it will be certainly adopted permanently among the powder classification systems used for industrial production mainly for its high throughput capacity, cleanness, very limited maintenance and total absence of moving parts and meshes that normally get clogged soon or later.

The drawback of the PI is in the limits in particle size resolution that hinders the adoption of the EJAC as it is with the current design. But its effectiveness in separating bigger particles (500-150 range) and medium sized particles (150-50) from the smaller ones is of great advantage in facilitating the overall sieving step, and substituting the air-classification for particles below 10µm.

The system throughput has been tested with different powders and fully satisfies the requirements. In the current installation the feeder has been tested with WCCo powder, with particles <500µm, able was able to deliver from 21 to 38 kg per hour. The Throughput capacity were actually limited by the coaxial venturi used to accelerate and inject the particles in the classification chamber, therefore throughout in the range of 20 to 80 kg/h are expected with small hardware upgrades.

The capital expenditure cost of the realized EJAC are in line with those of sieving and classifying technologies utilised for the same purpose, while the operative costs has to be carefully evaluated considering the manpower costs and the benefits from the reduced HSE issues: As evidenced by the histogram graph (Figure 4), although the overall energy consumption is much higher with the new PI solution (about +60%) there is a substantial decrease in manpower needed to operate the plant (about -85%).

In the pie chart of Figure 4 is reported the contribution of the different components of the EJAC to the electrical consumption: in this initial design the Air Blowers are the components that lead the overall PI energy consumption, but those are also the components for which there is more possibilities for optimization.

This estimation of operative costs does not include the maintenance costs, that are more difficult to estimate for the new PI although the new PI is envisaged to required less maintenance, due to the absence of moving parts as well as the absence of filters/meshes that normally have to be periodically substituted and disposed of.

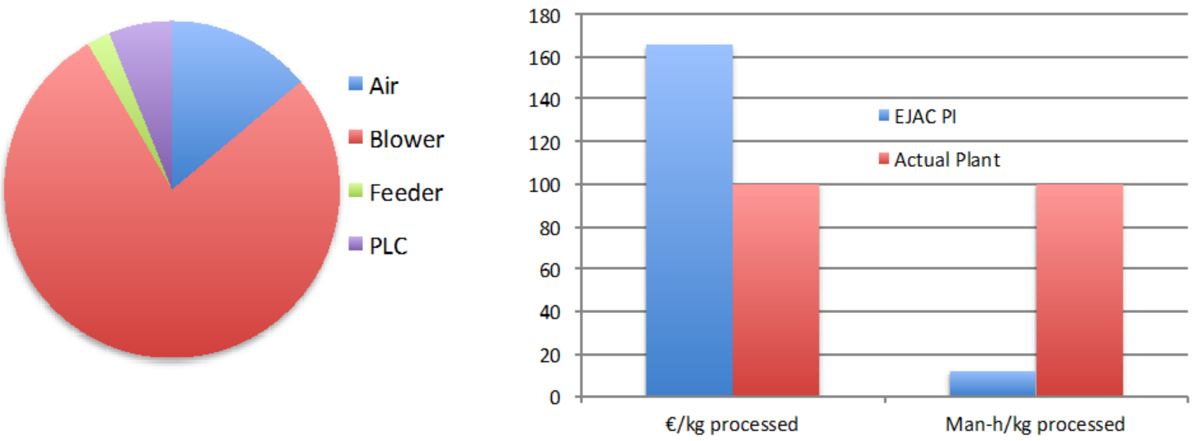


Figure 4. On the left: contribution of the different component of the EJAC to the overall electricity consumption, on the right a comparison with current solution of classification and sieving

TRL of PI Technology:

The Elbow-Jet-Air-Classifier installed in MBN nanomaterialia is in use for the classification of Tungsten-Carbide Cobalt grit obtained from the recycling of CerMet cutting tips by ball milling, the prototype is operative and its functionalities have been demonstrated in the operational environment. The EJAC can be considered TRL 7 for the multiple classification of ceramic and metal powders.

3. CS2: Ceramic Powder Processing

Case study host:

Euroatomizado, Onda, SPAIN

Case study leader:

Newcastle University, Newcastle, UK

Case study team:

Newcastle University, Newcastle, UK

Euroatomizado, Onda, SPAIN

ITC, Castelló, Spain

Analisis DC, Madrid, SPAIN

David Reay & Associates, Newcastle, UK

Brief description of process unit(s) of interest for intensification and motivation:

This case study has been focused on the intensification of the free-flowing powder preparation process involved in the very beginning of the ceramic tile manufacturing process. Nowadays, the most widely used ceramic tile shaping method is uniaxial pressing of spray-dried powder in hydraulic presses .The base case involves a wet route procedure where the initial free-flowing powder is obtained from a high solid content slurry, which itself produced by wet grinding of a mix of different solid raw materials with water and additive in continuous or discontinuous mills.

A free-flowing powder with a certain granule size distribution is required to assure a good distribution of the powder during the filling of press cavities. This is achieved in a spray dryer. Indeed, a heterogeneous distribution of the powder would cause different kinds of defects after firing e.g. differences in final tiles size, lack of orthogonality, or product deformations.

Together with the free-flowing character of the resulting powder, spray drying is also beneficial for the ceramic process because of the highly homogeneous final composition of the powder obtained by wet milling of solid raw materials. This is of major importance to dilute the possible impurities and thus lower their negative influence in the final product.

Spray-drying is proven to be a capable technology allowing one to obtain a granulated powder, the physical properties of which are specially adapted to the shaping of ceramic tile bodies by uniaxial pressing. Together with its homogeneous chemical composition, the spray dried powder shows four main properties which are key during the ultimate shaping stage:

1. Moisture content of 5-7 %: ideal for developing the plasticity of the clay fractions contained in the raw materials mixes.

2. The granulated powder obtained by spray-drying shows an almost spherical shape with very smooth surfaces.
3. The free-flowing spray died powder has a very narrow grain size distribution containing agglomerates from 125 to 1000 microns in size.
4. The spray dried granules are hollow inside because of the physical phenomenon involved in their formation.

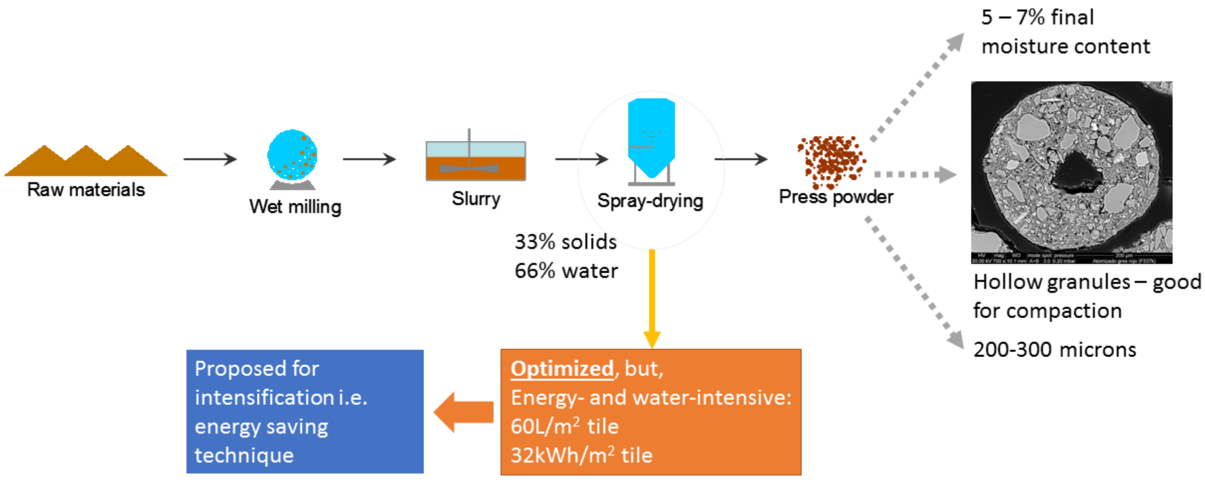


Figure 5. General flowsheet of the spray drying process in ceramic processing, which forms the basis behind case study CS2.

The primary objective of the case study was testing several dry route processing as an energy-efficient alternative to the established wet route processing, as shown in figure 6:

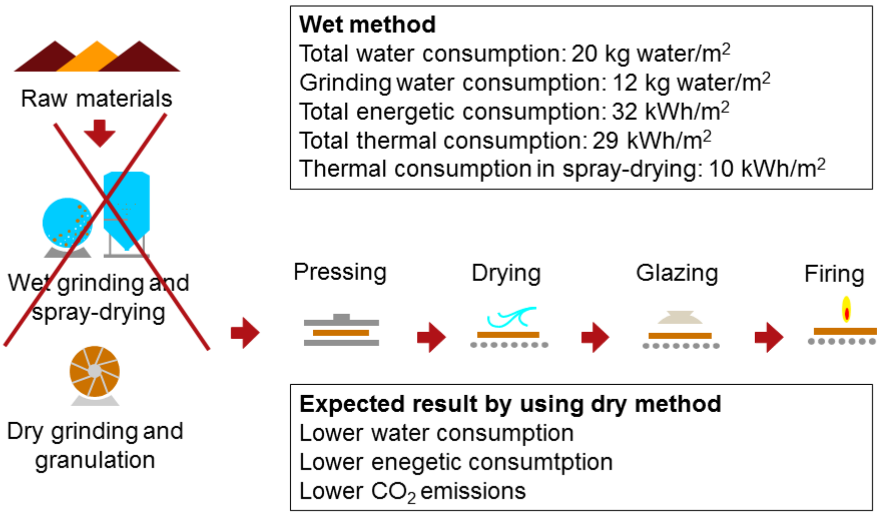


Figure 6. The merits of the dry route process. These are related to its energy and water savings.

Tests have not only included the validation of the intensified routes to produce a free-flowing powder, but also pilot tests to verify its behavior in the whole ceramic tile manufacturing process. And thus, to confirm that the physical and chemical properties of the final product prepared from the new powders are in accordance with the quality standards of the market.

Two technologies have been evaluated. On the one side, a direct high-shear mixing granulation in which a free-flowing powder is directly produced by agglomeration of the dry powder provided by a pendulum mill, using water as agglomeration agent. And on the other side, a rolling compaction methodology in which the dry powder provided by a pendulum mill is compacted by a pelletizer system and then the resulting pellets are grinded to obtain a powder with the desired particle size distribution.

The main difference between the two evaluated technologies is that after granulation, free-flowing powder needs to be dried to achieve the desired working moisture content. This post-process drying is not necessary when using the rolling compaction approach as the resulting powder contains the right amount of water.

Brief description of PI technology chosen:

As explained, two process intensification technologies have been chosen and proved during the development of the project: high-shear granulation and rolling compaction. Both technologies have been used to process dry powders provided by a pendulum mill. In both proposed dry routes, the pendulum mill would define the particle size distribution of the primary powder particles. By processing these primary powders, the process intensification technologies have been tested, adapted and optimised to obtain free-flowing powders with the best possible physical properties. The free-flowing powder physical properties considered during the technologies optimization have been the granule size distribution, the moisture content, the shape and strength of the granules and the flowability of the powder.

The high shear mixer-granulator is an established technology which has been retained from a KBE analyses conducted together with the different partners involved in the case study. Its highlight is that the granules are less spherical and denser, which presented lower deformation capability during tile pressing.

Together with Offenburg University staff, and other IbD partners, different workshops and working sessions have been conducted to define, by means of a TRIZ approach, new possible intensified technologies for the case study. By defining the problems associated with the case study functional analysis, different potential solution concepts were generated at the last workshop. And for this solution concepts the most suited technologies were the following ones:

- High shear granulation process:
- Roller compactor for granulation
- Screw granulation

Two of the proposed technologies suited for the detected solution concepts were already present in the KBE list of promising technologies, concretely, the high shear granulation and the screw granulation. The third one, the roller compacting technology for granulation, showed a good potentiality to be used as intensified technology. After some tests were performed with good results in one small scale facility located in Manfredini & Schianchi Srl site, in Sassuolo (Italy), further investigation was decided to be conducted in a complete pilot facility.

Granulation is a process involving particle size enlargement and is a complex process controlled by various mechanisms such us: wetting, nucleation, agglomeration and consolidation, breakage/attrition and layering. The final characteristics of the obtained granulates depend on the equipment design. The equipment used in this demonstrator is the special EIRICH mixing system comprising 3 main components. The way in which these components are used can be varied in a particularly flexible mode when designing the mixing processes.

- The rotating mixing pan, which delivers the mixture into the area of the mixing tools
- One or more mixing tools arranged eccentrically. The direction of rotation and the speed of the mixing tool(s) can be optimally adapted to the different applications.
- The bottom/wall scraper, providing additional agitation action. It prevents caking on the wall and bottom of the pan and facilitates discharge when the mixing cycle is complete.

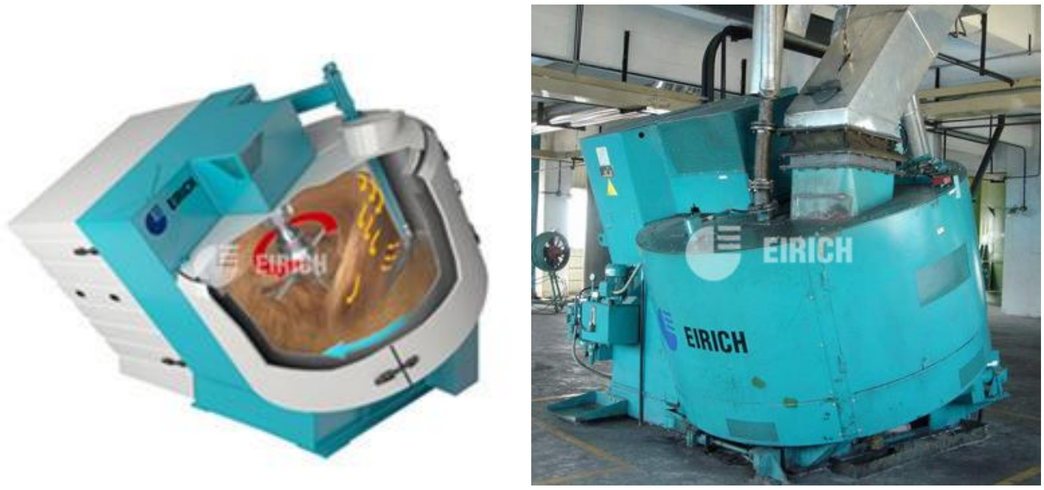


Figure 7. Eirich granulation-mixing technologies for dry granulation of ceramic powders (left: batch mixers, right: continuous mixer).

Concerning the **rolling compaction technology,** a pilot facility provided by the Italian company Manfredini & Schianchi was installed in Euroatomizado's site. This facility was based in the Fusion technology developed by Manfredini and it has been used to verify the preliminary results obtained in several tests conducted in the supplier facilities. Using the Manfredini roller system based on the principle of press-agglomeration, particulate material is introduced into the nip of the two counter-rotating rollers by means of vertical feeders. As the material was compacted, pressure within the material increases, reaching a peak just above the line of closest approach between the rollers, and subsequently drops rapidly to zero. During this process, the apparent density of the mixture increases by a factor of 1.5 to 3 due the decreasing void volume of the bulk material. The resulting product is typically a pelletized material as the one shown in figure 8.

The pelletized material produced in the roller press should be broken up into granules and classified into the desired particle size range. Often, the first crushing step takes place in a specially designed flake breaker installed directly below the roller press. The pellets were broken into smaller pieces (flakes) which make perfect feed for subsequent crushers.

Using a multi-deck screen, the material was then separated into oversize, product and undersize classifications. The undersize (which passes directly through to the lowest screen deck) was re-circulated for compaction, while the oversize formed a residue on the upper screen deck which was fed back into the size reduction process.

Figure 8. Pelletized material resulting from the rolling compaction stage of the Manfredini Fusion system.

The main advantage of this system is that the quantity of water which is required for powder pelletizing is the same as the one required in the final product. Because of this, it is not required to use a big amount of water to guarantee a good granulation of the powder and no ulterior drying operations would be required. However, during pilot testing, special efforts have been conducted to achieve a homogenous shape in the final granulates as, by defect, they show irregular shapes after the crushing stages conducted after pelletizing.

Different pictures of the pilot rolling-compaction facility deployed at Euroatomizado's site are shown. On the one hand the pelletizer and the crushing equipment, and on the other hand, a detail of the pelletizing molds.

Figure 9. Pictures of the rolling-compaction pilot facility.

Different pictures of the high shear granulation equipment are presented below:

Figure 10. High shear mixing equipment.

Brief summary of results:

Main results obtained for both technologies are summarized below:

- **Granulation:**
    - Good granule size distributions could be obtained with the tested technology. The resulting granules sizes are like the ones obtained by the wet non-intensified technology. Moreover, the working parameters to control these distributions have been identified and studied. The evolution of the granules size during the process have been determined and used to validate simulation models

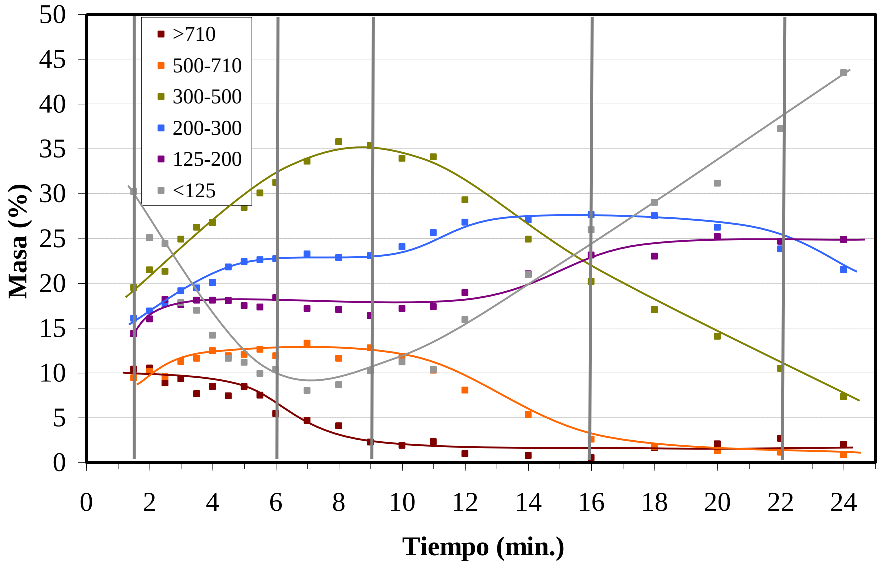


Figure 11. Evolution of granule size fractions with granulation time at variable granule moisture contents.

- The evolution of granule bed density and of granule hardness with granulation time was determined. The hardness of the granules obtained with the high-shear granulator was greater than that of material obtained by spray drying, after stirring times above 2 minutes.

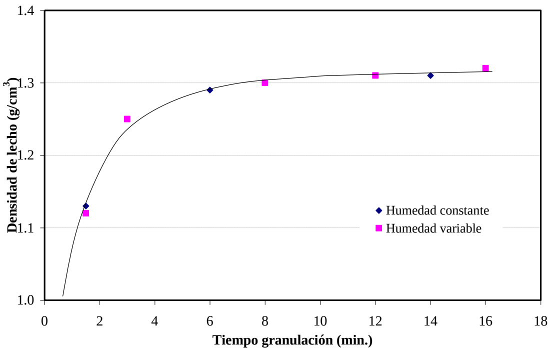


Figure 12. Bed density

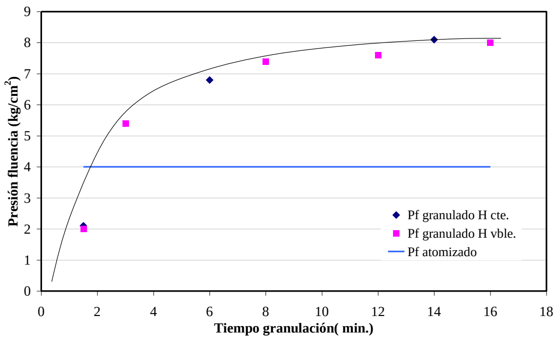


Figure 13. Yield pressure

- Properties of the resulting tiles shaped by using granulated powders are similar to the ones obtained with spray-dried powders. The main disadvantage of the technology is that a post-drying is necessary to condition the moisture content of the powders after granulation.

- **Rolling compaction:**

  - The properties of the resulting tiles obtained with the free-flowing powders provided with this technology are very good and not moisture conditioning is necessary after the process.

	Intensified Technology	Standard spray-drying
Powder moisture content (%)	6,5	6,5
Dry bulk density (kg/m^3)	2079	2160
Dry Mechanical Strength (kg/m^2)	27,5	26,4
Linear Shrinkage (%)	-0,75	-0,56
Fired Bulk Density (kg/m^3)	1761	1831
Water Absorption (%)	18,70	16,44
Fired Mechanical Strength (kg/m^2)	23,8	23,6

  - The technology needs to be improved by using other grinding technologies to provide a better control of the final granule size distribution.

Final conclusions from case-study:

The main conclusions drawn from the validations and works shown on this report are as follows:

- Concerning the **high shear mixing technology**, the water addition method did not influence granule size distribution. When the agglomerate moisture content remained constant, the growth mechanism prevailed throughout the test. The granule size fraction between 300 and 500 µm maximised at about 8 minutes. This maximum practically coincided with the minimum of the fraction below 125 µm under the tested conditions. This granule size curves are like that obtained with spray drying base case.

- Regarding the **rolling compaction technology**, the grain size distribution which could be obtained is not so good as the one obtained with the high shear mixer granulator. The conducted tests shown that with the current range of variation in the working parameters it is difficult to obtain final powders with a grain distribution curve like the one obtained by spray drying. Results evidence that further development in the grinding technology should be done to reduce the thinner fractions of grains. Anyway, the resulting powders have shown a very good ceramic behaviour and research on alternative grinding technology have been started with very good preliminary results.

- TRIZ methodology has been useful to identify alternative PI technologies. The most promising results have been obtained with the rolling compaction technology which has been proposed from a the TRIZ workshop sessions.

- All the evaluated technologies involve a dramatic reduction of energy and water consumption. From this point of view, the rolling compaction technology linked, with an appropriate grinding technology is the most suitable technology, as it allows a powder preparation with a restricted use of water. High shear-mixing technology requires using higher amounts of water which lately need to be removed by drying.

TRL of PI Technology:

Both proven technologies can now be considered TRL 7-8 for manufacturing free flowing powder for ceramic tile manufacturing.

4. CS3: Mineral Beneficiation

Case study host:

Pyhasalmi Mine Oy, Pyhäsalmi, Finland.

Outotec Oyj, Espoo, Finland.

Case study leader:

Outotec Oyj, Espoo, Finland.

Case study team:

University of Oulu, Oulu, Finland.

VTT Technical Research Center of Finland, Oulu, Finland.

Dynamic & Security Computations S.L. Analisis-DSC, Madrid, Spain.

Brief description of process unit(s) of interest for intensification and motivation:

Mineral beneficiation processes chain incorporate several unit operations operated in continuous manner. In the crushing line the solid material (typically ore) is comminuted to the particle size of centimetre scale. In grinding line, water is added and the particles are processed further into micrometer scale using grinding mills. In this stage, the particles are classified and directed to downstream processes. Finally, the valuable material is recovered in flotation line, where chemicals and physical phenomena are utilized to separate the concentrate from the gangue.

Whilst the high throughput and very energy intensive process chain may be difficult to be intensified via smaller equipment, new process equipment and better understanding of the process state can support the PI targets. In the mineral beneficiation process chain intensification demonstration, several approaches are taken to avoid excess use and loss of energy in the grinding and flotation sections.

The simplified process chart for the grinding and the flotation circuits of Pyhasalmi mine are given in Figure 14, also showing the unit processes covered by the Oulu Mining School Minipilot facility (University of Oulu). The OMS Minipilot facility was developed based on the beneficiation plant of Pyhäsalmi mine with the scaling ratio of 1:5000.

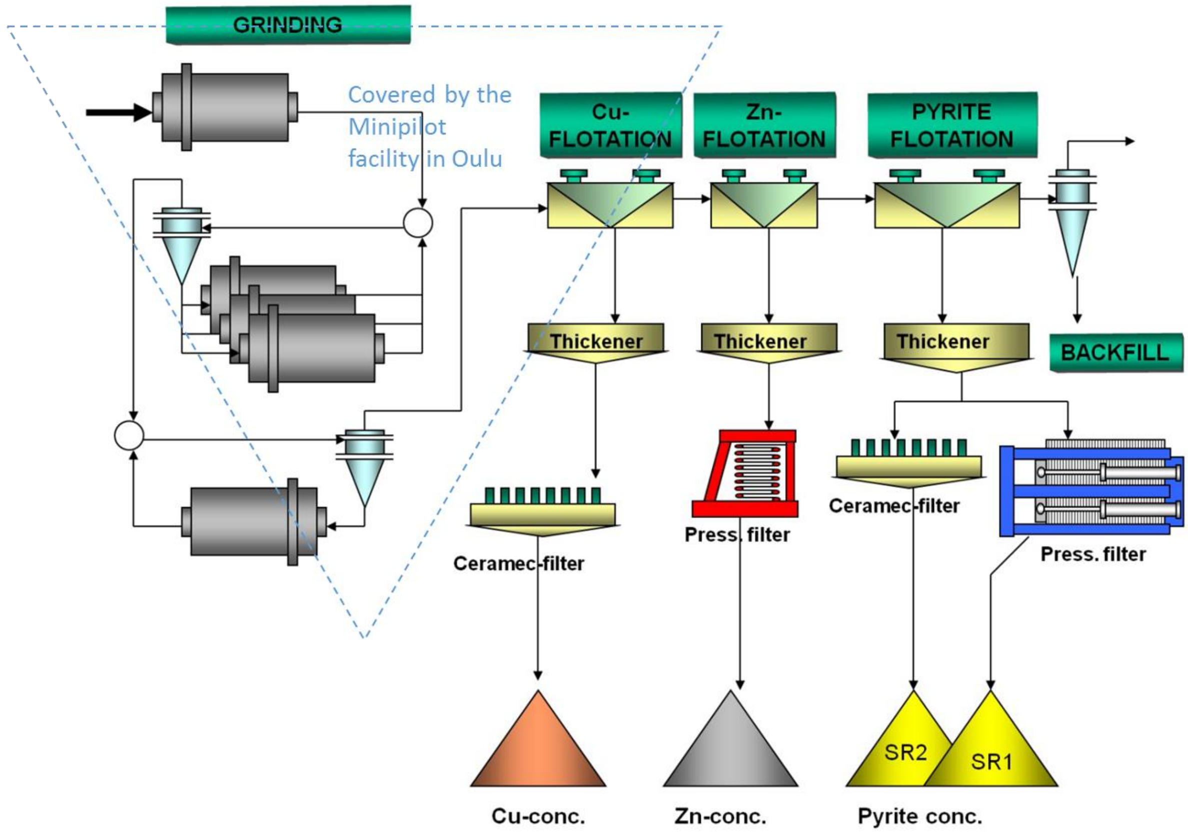


Figure 14. Process description of the Pyhäsalmi concentrator plant.

Brief description of PI technology chosen:

The intensification in this case study utilized several PI technologies; a coarse flotation device (or flash flotation), model predictive control (PAT tool), Raman spectrometer, bubble size measurement, particle tracking measurement and electrical resistance tomography (PAT tools), and dynamic modeling and adaptation (PAT tools).

The coarse flotation testing was concentrated on small scale coarse flotation device, shown in Figure 15, installed to the OMS Minipilot facility. The coarse flotation device consists of cell with monitoring window and three probe orifices/valves aside in order to support the testing of new measurement technologies. In coarse flotation tests in the mini-pilot, the grinding circuit was operated in different intensities to produce different kind of feeds, in terms of slurry density and particle size, for the small scale coarse flotation machine. The outputs from the small scale coarse flotation machine tests are utilized in the development of a phenomenological model for the machine.

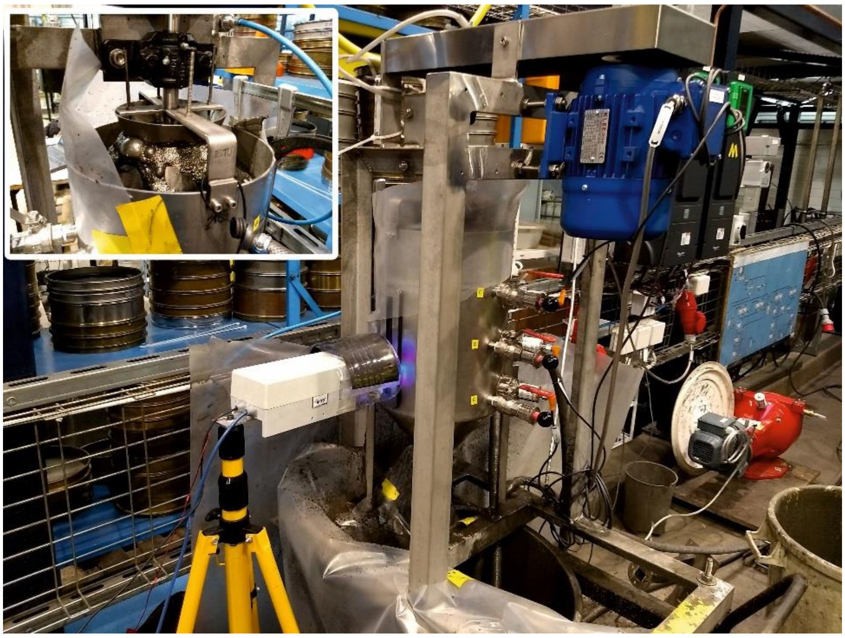


Figure 15. Coarse flotation device in minipilot-scale. On top, a copper concentrate is flowing from the top of the device. In the lower left corner, the particle tracking measurement device is also seen.

The Raman spectrometer has been tested in the coarse flotation machine utilizing the tailored sample orifices of the machine. The test arrangement is depicted in Figure 16. Raman spectroscopy offers a direct way to measure the mineral concentration in-line. This measurement can potentially be used in the control of mineral beneficiation, which would then have an effect to economic, energetic and environmental performance of the plant. The Raman measurement was also tested in industrial environment, where the probe was connected to Courier XRF instrument in Pyhäsalmi mine. The bubble size measurement prototype was tested in Pyhäsalmi mine, where it was directly submerged into a conventional flotation cell, as seen in Figure 17.

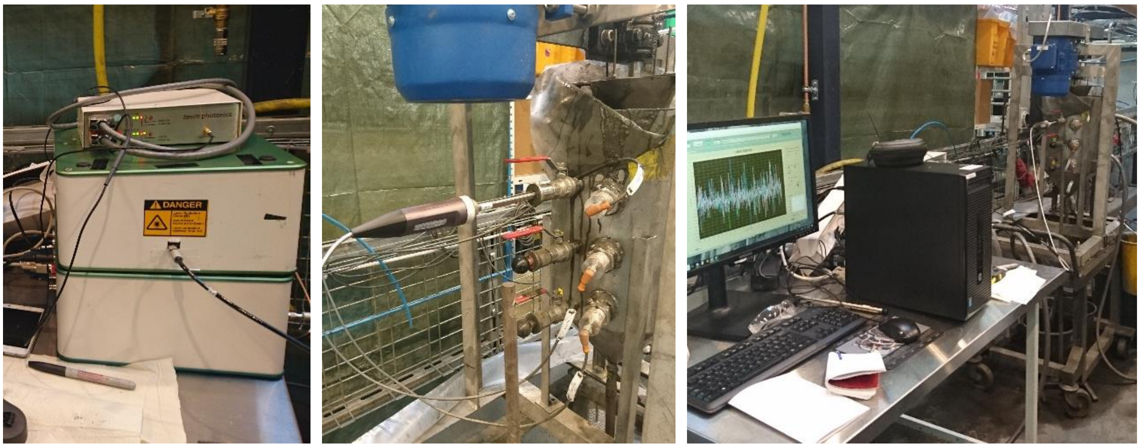


Figure 16. a) TimeGated Raman instrument M1, b) customized Marqmetrix Performance Ball Raman Probe connected to coarse flotation cell and c) TimeGated instrument PC connected to the M1 spectrometer next to the flotation cell.

Figure 17. a) Measurement device: a machine vision camera with a ring of white LEDs around the lens inside a long waterproof housing; b) Measurement device inserted into the flotation cell through a hole in the grate.

The new solids measurement technologies may offer additional ways to monitor the process streams in grinding and flotation circuits. In addition to on-line measurements, utilizing indirect measurements to provide information about unmeasured variables would allow to monitor the state of the system and facilitate better usage of resources trough a plant-wide decision support system (DSS). As a part of this demonstration, a dynamic mass balancing module was implemented to Pyhasalmi mine. Data reconciliation algorithms utilize known process and measurement variations, namely standard deviations. They will assist the computation algorithm, and determine whether each measurement should be adjusted to hold the mass balance over all the process units in the plant. Calculations are typically based on flowsheet models. In this case, the on-line calculation routine is based on Outotec's Advanced Control Tools (ACT®) and HSC® Chemistry Software. In order to run such a dynamic simulation model with reliable results in changing process conditions, an online adaptation algorithm was developed during the project. The model adaptation scheme, shown in Figure 18, continuously updates the flowsheet model with the measured data from the real process.

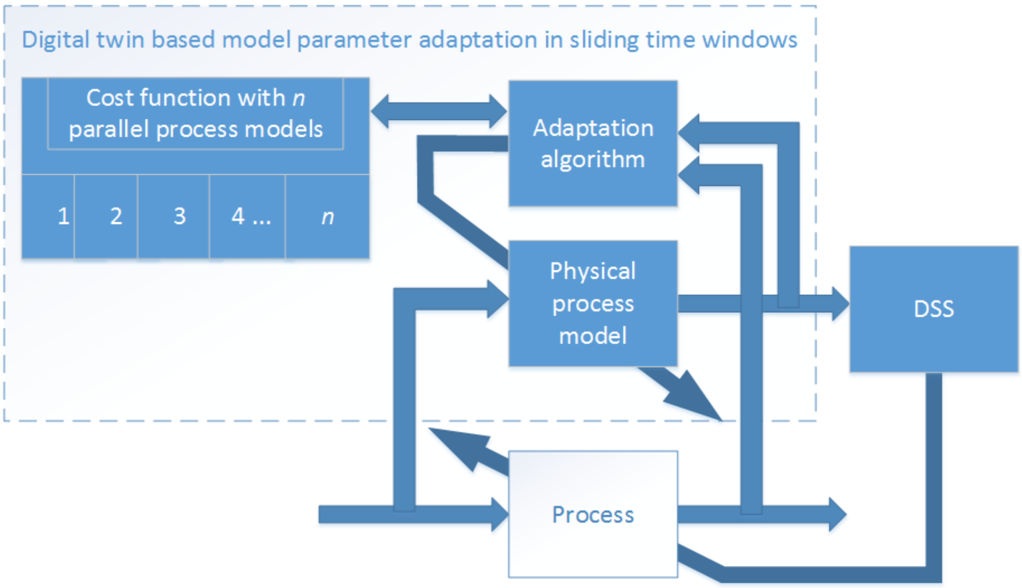


Figure 18. Resulted model parameter adaptation scheme**.**

Brief summary of results:

The effect of the coarse flotation in different possible locations within the grinding circuit was evaluated with mass balance calculations. Although there are number of successful implementations of coarse flotation machine world-wide, the pilot scale coarse flotation tests with Pyhäsalmi ore did not support the PI installation in terms of energy savings in grinding circuit. However, the 'hot float' experiment and modeling with another ore type indicated the feasibility of the PI technology in terms of increased copper recovery. According to these results, a greater impact could be achieved by installing the device to a non-conventional location in the process.

The advanced grinding circuit control has been successfully implemented and tested. It seems that the Model Predictive Control (MPC) can improve especially the primary mill charge level and stabilizes particle size distribution (PSD) for the grinding circuit product, and therefore results as a more stable and robust process against fresh ore feed variations. Quantitative results and evaluation of the final impact of this PI solution will be available later once formal ON/OFF trials have been completed.

The mineral content measurement with Raman spectrometry shows a great potential for introducing new on-line mineral analysis capabilities. The Raman measurement could be used in closed-loop control of mineral beneficiation plant, but robust measurement heads or probes to an industrial analyzer need to be developed and the applicability of Raman measurement for different mineralogy needs to be studied.

Bubble size measurement tests indicate that the prototype can be used to successfully measure the bubble size distribution inside a flotation cell. The validation process is still ongoing, but the initial results seem promising. The method could be used to provide on-line bubble size measurements.

The results of the particle tracking tests were conflicting. While the vector field directions were consistent across measurements, the actual flow velocity measurements did not correspond. Further development is needed to make the method effective.

The dynamic mass balance module has been running in Pyhäsalmi mine for several months. Although the complexity of the flowsheet doesn't allow to take the full advantage of dynamic mass balancing without additional measurement instrumentation, it can already offer new information on one flotation stream. This information can be important in understanding the process state and recovering from abnormal situations. The model adaptation scheme developed makes possible to track the changes in the process real-time and can be used as a soft sensor for variables that are difficult or expensive to measure including e.g. the raw material variations. The initial results, shown in Table 1, indicate that in a short time window the flotation parameters could be estimated accurately and the algorithm is likely to meet the soft sensor requirements regarding to relative modelling error.

Table 1. Parameter search space and adaptation result.

Parameter	1	2	3	4
Lower limit	1.4	0.1	0.005	0.0001
Upper limit	3.4	0.5	0.45	0.01
Real value	2.0	0.3	0.02	0.001
Adapted	2.0	0.3499	0.021	0.001

Final conclusions from case-study:

- The case study has resulted as two PAT methods successfully implemented to Pyhäsalmi concentrator plant.
- The quantified process intensification impact can only be seen after longer period of operation than available during the project timeline.
- Several other PAT methods were also demonstrated. The results are encouraging especially regarding to the novel optical measurements and on-line model adaptation. However, further testing and development is required before productization or online implementation.
- The coarse flotation is a proven technology, but the performance in minipilot scale didn't support the full-scale implementation with the studied ore.
- The results indicated that a greater impact with coarse flotation could be achieved by installing the device to a non-conventional location in the process.

TRL of PI Technology:

The TRL of the used PI technologies are presented in Table 2.

Table 2. TRL evaluation of the demonstration activities.

	Coarse flotation	**Advanced control**	**New measurements**	**Dynamic mass balancing**
TRL	5*	7	4-7**	8

*TRL applied to the analysis of the performance of the coarse flotation machine in a new type of flowsheet configuration. Flash flotation cell itself is proven technology (TRL 9).

**ERT flow measurement validated in laboratory (TRL 4). Particle tracking demonstrated in pilot-scale (TRL 5). Raman and bubble size measurements tested in industrial environment (TRL 7).

5. CS4.1: Pharmaceutical Processing I

Case study host:

Newcastle University, Newcastle-upon Tyne, United Kingdom.

Case study leader:

Newcastle University, Newcastle-upon Tyne, United Kingdom.

Case study team:

Sanofi-Aventis, Barcelona, Spain.

Newcastle University, Newcastle-upon Tyne, United Kingdom.

FreemanTech, Tawkesbury, United Kingdom.

Analisis-DSC, Madrid, Spain.

Brief description of process unit(s) of interest for intensification and motivation:

Sanofi-Aventis in Spain is manufacturing the proprietary L-thyroxine tablets (a hormonal drug) with a common granulation equipment used in pharmaceutical industry: a high shear mixer, running in batch-wise operation. In this case study, we examined the efficacy of continuous granulation for this drug formula by using a twin screw granulator operating in continuous mode.

Granulation is a size enlargement process where particles are brought together to form larger permanent agglomerates. Granulation improves the physical properties of a material making it easier for handling and downstream processing. In pharmaceutical industry, the granules are typically used as an intermediary before compaction into tablets, the most common type of oral solid dosage.

In 2004, the US Food and Drug Administration launched the process analytical technology (PAT) concept to stimulate the pharmaceutical industry to change from off-line to real-time quality testing of material properties (Plumb, 2005, https://doi.org/10.1205/cherd.04359). This way, material quality can be maintained in the production line, eliminating the variation in products. This is common in batch manufacturing. In addition, to scale-up the production of granules, the twin screw granulator can simply be run for a longer period of time. Production scale-up in batch however, often requires bigger, more expensive equipment and time-consuming. These and other factors have shifted the mindset of pharmaceutical giants to switch from the conventional batch to continuous processing.

According to Fonteyne et al., 2015 (https://doi.org/10.1016/j.trac.2015.01.011), the entire pharmaceutical manufacturing process can be continuously operated, from mixing of initial powders to packaging of final tablets. This can be done with a twin-screw granulator and the subsequent drying in a fluidized bed system as its core processes. The viability of twin screw granulation in particular, depends on its delivery of consistent and optimum granule properties, mainly in terms of particle size distribution, bulk density and flowability. The correlation between screw design, processing conditions and granule properties forms the research scope of this case study, specific to the L-thyroxine granule production.

Brief description of PI technology chosen:

Drug formula: The recipe involved two types of starches and one type of cellulose crystals as the excipients. Excipients are the bulking agents for tablets. In practice, trace amounts of active pharmaceutical ingredients (APIs) are added. However, in these tests, the APIs were not included as we were only examining the physical attributes of the granules. The granulation liquid was mainly water with some dissolved binding agents.

The twin screw granulator (Figure 19): Located at Baker Perkins, Peterborough (United Kingdom), the pilot-scale equipment (24 mm diameter, 600 mm length screws) was used for the granulation tests. The excipients were pre-mixed and fed into a hopper, where automated control of valves ensured precision in powder flow rates. The equipment was pre-programmed with the desired material flow rates, screw speeds and barrel temperatures: based on pre-planned design of experiments. This ensured accurate testing protocol with efficient costs in time, man-hour and material. The residence times for granulation were of the order of seconds and the collected granules were then subsequently dried and analysed for their physical properties. Scanning electron microscopy (SEM) also allowed a close examination of the granules.

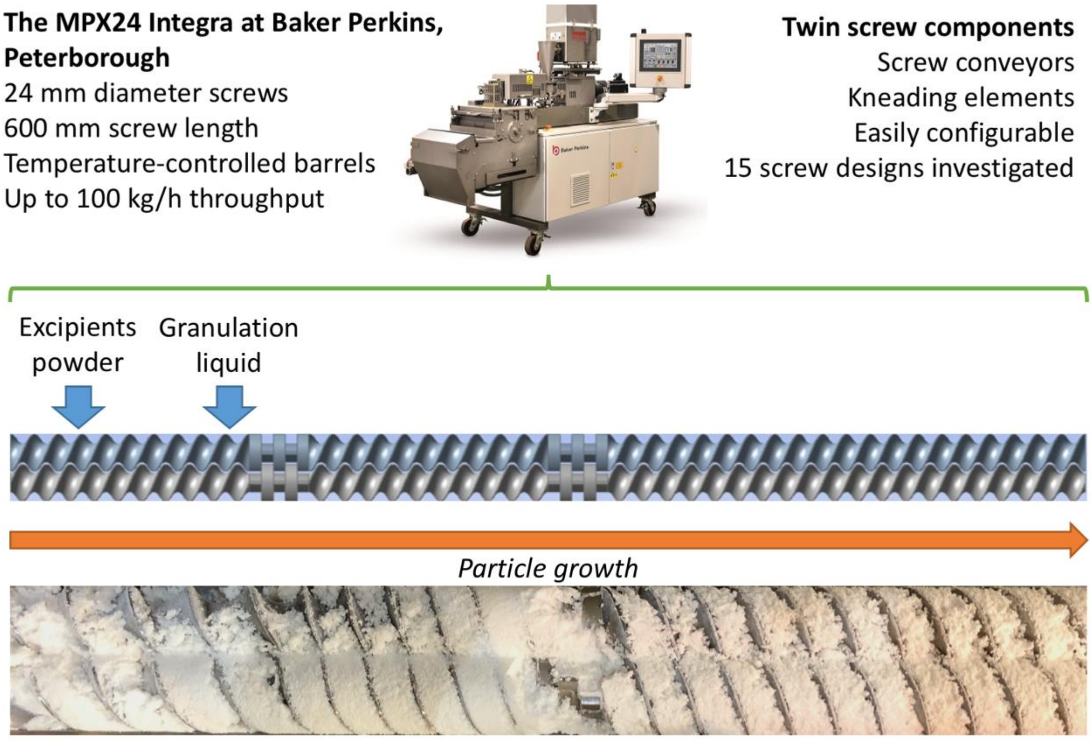


Figure 19. The twin screw granulator. The twin screw design shown was only a representative design out of fifteen designs investigated.

Brief summary of results:

The aim of the tests was to reproduce as closely as possible the granule consistency obtained in current batch processing in terms of optimal particle size distribution and bulk density, by carrying out the granulation process in the twin screw granulator in continuous mode of operation. Various screw designs and operational conditions such as liquid flow rate and screw speed were adjusted to investigate their effects on the granule properties of interest. Guidelines for the desired granules were: small size (< 150 microns) and high density (0.5 – 0.6 g/mL) granules. These were to accommodate the relatively small tablet size of L-thyroxine drugs and to secure the valuable API particles in the granules.

Results have shown us that parametric changes imparted significant effects in both granule size and bulk density. Higher liquid flow rates produced larger granules due to bigger nuclei formation as more fluid was available. Larger granules were also produced via reducing screw speeds as the levels of barrel fill were increased, resulting in higher compaction probabilities among particles. The interplay between screw design and particle properties was also found to be substantial. Shown in Figure 20, screw designs with kneading elements produced good bulk density of ~0.56 g/mL, but with high percentage of oversized (> 1 mm) granules. Whereas screw designs without kneading elements produced granules with opposing properties: good particle size distribution but, with frail and porous granules.

The example data presented here indicates how finding an optimal design and process conditions can be challenging when multi-variables were involved, most importantly: screw design, barrel temperature, powder and liquid flow rates. The tests carried out have successfully narrowed the design space of several parameters, which information can be used for further research. Also, the tests have confirmed the viability in producing the desired granule properties, carried out in continuous production via the twin screw granulator.

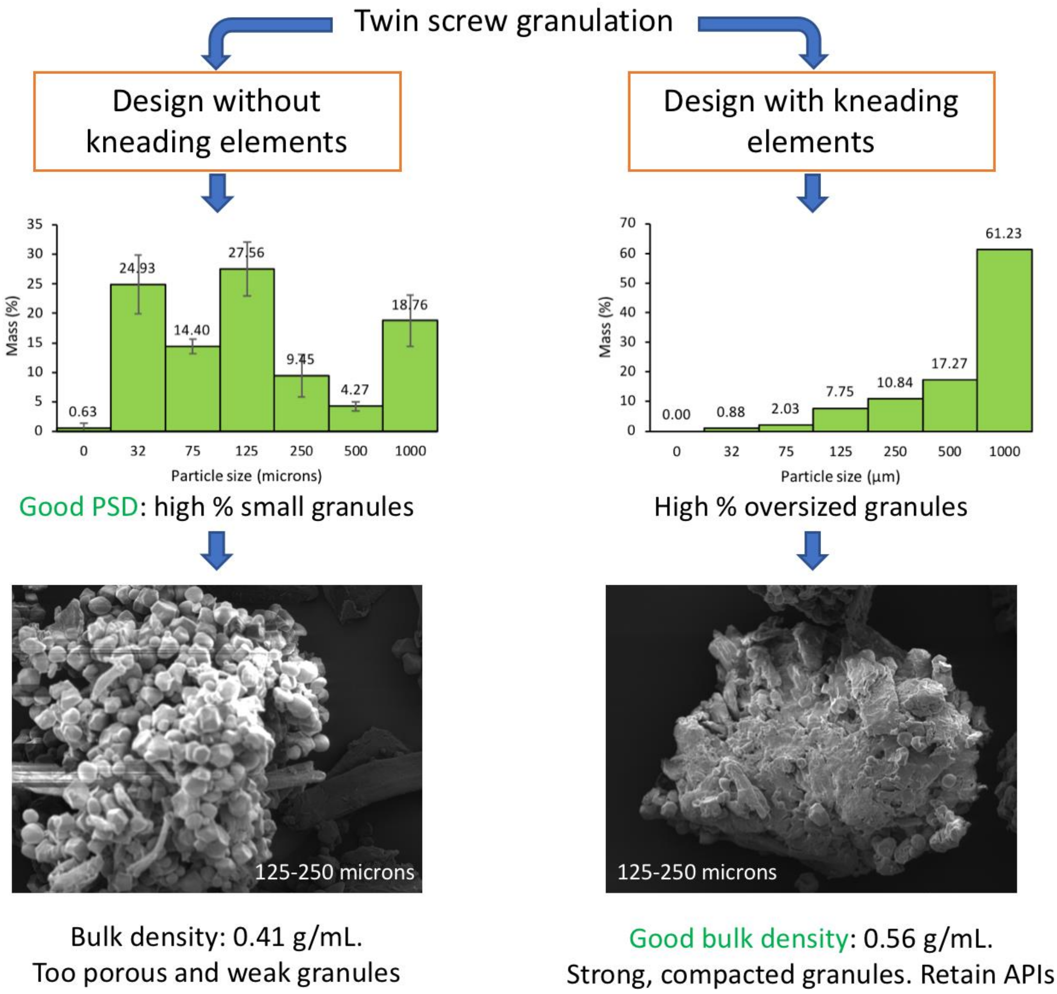


Figure 20. The effect of screw designs (e.g. with/without kneading elements) on granule properties was found to be substantial.

Final conclusions from case-study:

The tests have demonstrated the viability of the twin screw granulator in the continuous production of granules for the L-thyroxine drug tablets, as opposed to the current batch production. However, further research into the optimal screw designs and process conditions is required for full economic and technical assessments of this technology. The tests have also indicated that overall, the energy consumption per kg material processed is very competitive at 0.04 kWh/kg in the continuous twin screw granulator. Due to the self-wiping profile of the screws, fouling issue was found to be negligible and this was important to ensure an uninterrupted continuous granulation.

TRL of PI Technology:

The twin screw granulation can now be considered as TRL 6 (technology demonstration level) for pharmaceutical granulation.

6. CS4.2: Pharmaceutical Processing II

Case study host:

Industrias Farmacéuticas Almirall, S.A., Sant Andreu de la Barca, Spain

Case study leader:

Eindhoven University of Technology, Eindhoven, The Netherlands

Case study team:

Analisis DSC, Madrid, Spain

Freeman Technology, Tewkesbury, United Kingdom

Zurich University of Applied Sciences, Zurich, Switzerland

Leitat, Terrassa, Spain

Brief description of process unit(s) of interest for intensification and motivation:

The Almirall case study, CS4.2, concerns drying of a wet granulated material including a binder solution (purified water + povidone), that is currently dried in a multi-stage vibrating fluid bed dryer (Heinen).

The large production volume and energy use from this part of the process are most promising for further optimization and have ample opportunities to benefit from intensification.

Brief description of PI technology chosen:

In order to minimize energy use and keep residence time within limits, a very suitable option was the spiral flash dryer (SFD). This technology acts as a dryer and separator at the same time, keeping wet particles still inside the dryer whereas dried particles are separated. The use of drying air is minimal, and the equipment size can be reduced, bringing further cost reduction.

Brief summary of results:

As for the implementation of the concept into the existing process, from an industrial perspective, the comparison of the existing HEINEN dryer and the SFD pilot dryer are summarized as follows:

Current process (HEINEN DRYER).	Differences detected with new TORBED machine.
Inlet air supply and blow air supply.	No critical changes compared to the current equipment
Dryer machine Size.	Considerable reduction in the size of the equipment.
Cylindrical Outlet filters.	The design of the filtration system by means of cyclones and filters increases significantly the size of the equipment layout . This topic introduces a critical point to discuss .

Indeed, the SFD provides a significantly smaller equipment size, but the filtration system becomes larger and this largely defeats the purpose of the SFD, while the particle properties have different characteristics as measured (flow properties, granule size), as mentioned above. Also, there is no cleaning in place system according to the pharmaceutical standards and, moreover, setting up a new drying equipment comes with its own costs, such as variation in the Marketing Authorization Application and risks of losing years of experience with the current drying system; the Almagate product has production requirements and the risk of having less control over the process does not outweigh the potential benefits. These reasons alone already make it hard to justify an implementation of the SFD in the existing process.

Moreover, prior to additional processing, granule characterisation using the FT4 Powder Rheometer® demonstrated clear differences between samples of Almagate dried using the Heinen and TorBed dryers. Therefore, this analysis indicated that the use of different dryers had a measurable impact on the rheological properties of the Almagate granulate directly out of the dryer.

The technology is considered as a suitable technique for drying of powders and provides a novel route compared to classical drying equipment for granulate drying processes in general. Almirall has stated that, if a process would have to be designed from scratch for a new product, the SFD would be taken into consideration again.

Final conclusions from case-study:

The SFD drying concept should be considered for drying of granular materials for novel processes, not for replacement of existing processes. The control procedures may then be adequately adapted to the new process. Especially when a constant inlet quality is expected, the SFD concept may pay off due to the lower overall energy consumption. However, it should be taken into consideration that pilot trials are still required before implementation in industrial processes.

TRL of PI Technology:

The spiral flash dryer can now be considered TRL 6-7 for drying of wet granulate.

7. CS5: Intensification of Chemical Processing Involving Solid Reagents

Case study host:

University of Leeds, Leeds, UK.

Case study leader:

University of Leeds, Leeds, UK.

Case study team:

University of Leeds, Leeds, UK; AM Technology, Runcorn, UK.

Brief description of process unit(s) of interest for intensification and motivation:

Scale reduction is a key aspect of process intensification. The benefits can be related to enhanced performance of small equipment or more efficient use of space and is usually a combination of the two. The productive operating times of large batch reactors is generally less than 10%. A typical batch plant uses 10 to 20 batch reactors of different sizes to cope with variations in working volume requirements. A 10-litre flow reactor by comparison, can process 480 litres in a 24 hour cycle for a 30 minute reaction. The same reactor can also process from 1,000 or 100,000 litres in a single operating cycle depending on the number of reactors used in parallel. This efficiency means that 4 or 5 flow reactors of a single size can replace 10 or 20 large batch reactors of different sizes, which delivers substantial reductions in capital expenditure and operating costs. Given the significant advantages of flow reactors, there are strong drivers for industrial process intensification of flow systems. However, traditional (single-tube) plug-flow reactors have a critical downside, being the small residence time distributions for reactions requiring fast turbulent flows for good mixing. This limitation is particularly true for reactions involving solids, as plug flow reactors generally do not have the capacity to pump at speeds to keep solids suspended, leading to sedimentation and fouling at inlets/outlets.

Brief description of PI technology chosen:

The AMT *Coflore ATR* reactor (http://www.amtechuk.com/pilot-and-plant-scale-atr/), shown in Figure 21, is an agitated tubular reactor that provides a novel solution to the issue of mixing dynamics in plug-flow to enhance residence times. This solution decouples the plug flow and mixing from bulk flow, giving the ability to handle gases, liquids and solids: the *ATR* consists of a pneumatically driven main body that contains a number of reaction tubes into each of which a smaller, free-moving, perforated agitator tube (with plastic end-caps to minimise friction) is inserted. The lateral driving motion applied to the system results in efficient mechanical mixing, even in cases of slow bulk flow (leading to long resistance times and faster reaction kinetics). In this case study, the *ATR* is demonstrated as a novel intensified plug-flow reactor, suitable for a variety of solid-liquid chemical processing operations, with focus on intensification of solids catalysed chemical reactions. AMT's interest is strongly focussed towards more efficient use of equipment with improved performance as an added benefit.

In this case study, a 1 L pilot scale AMT *Coflore ATR* reactor is characterised, to better understand the mechanical motion and hydrodynamics, enabling optimisation of the reactor to conduct solids catalysed liquid chemical reactions. The associated Deliverable reports detail work that has been undertaken to commission and demonstrate the ATR reactor at the University of Leeds. It includes analysis of the reactor movement frequency versus amplitude relationships for different pneumatic drive pressures, using a laser displacement device, as well as the characterisation of the relative movement of the inner agitator in relation to the outer reactor tube, using high-speed video-image analysis. An ultrasonic velocity profiler (UVP) is used to provide velocity profiles of the fluid across the diameter of the outer (*i.e.* driven) tube, which is, to the authors knowledge, the first time such as system has been used to measure the complex flow patterns of such a complex process-intensified tube reactor. Physical characterisation is coupled with computational fluid dynamics (CFD) simulations of the full-scale ATR system, to provide a more complete understanding of the mixing hydrodynamics and mixing energy for different oscillation frequencies. Additionally, the influence micro-mixing and turbulence regimes on particle suspension is modelled using direct numerical simulation (DNS).

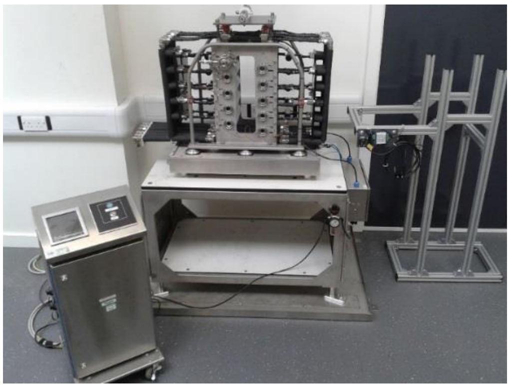
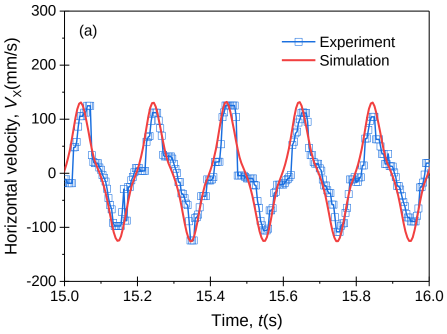
(a)  (b) 

Figure 21. (a) *ATR* in place at UNIVLEEDS, July 2017, without full diagnostic set-up. Laser displacement device shown on right of picture; (b) rendered image of the design of a perforated inner agitator tube used in experiments.

Brief summary of results:

A regime map of the motion of the agitator was derived using data from the laser displacement device in order to determine the optimal operating conditions. On the basis of the regime map, three test cases (in terms of applied pneumatic pressure, *p*, and therefore amplitude of motion, *a*, and agitation frequency, *f*) were chosen for more detailed experimental and computational investigation: (1, base) *f* = 5.00 Hz, *a* = 12.5; (2) *f* = 4.07 Hz, *a* 10.2 mm; and (3) *f* = 3.13 Hz, *a* = 11.2 mm. Selected results for one test case are shown in Figure 22 and Figure 23. Here, it is evident from Fig. 2, that CFD simulation and direct visualisation of the relative agitator motions are directly comparable, while measurements of the RMS of velocity (associated with turbulence modulation) with the UVP also correlated closely.

(a) 

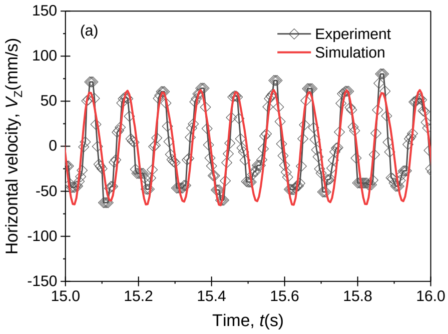
(b) 

Figure 22. Comparison between experimental video analysis and CFD simulation - relative velocity of ATR motion of internal agitator: (a) horizontal velocity, and (b) vertical velocity under shaking condition of Case 1 (*f* = 5 Hz, *A* = 12.5 mm).

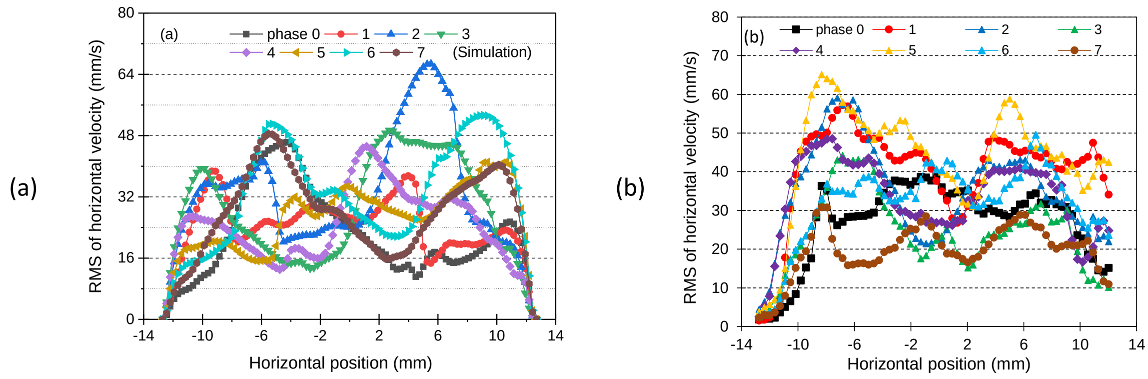


Figure 23. Phase-resolved RMS of horizontal (radial) fluid velocity under condition of Case 1 (*f* = 5 Hz). (a) CFD simulation, (b) experimental results via UVP.

Flow-field maps and power consumption calculations from the numerical model were also prepared for each of the three cases and are available in Deliverable report D6.3, but are not shown here for concision. The process-scale CFD predictions of particle mixing and sedimentation were also compared against direct numerical simulations of a simplified system with similar relative turbulence levels. Work additionally included understanding process chemistry kinetics and yield variation for a model solids catalysed reaction at various oscillation frequencies versus batch reactor results. Finally, a larger industrial scale unit (2x geometric size of the pilot-rig) was demonstrated at AMT, with process chemistry results compared to the pilot rig.

Final conclusions from case-study:

The motion of an agitated tube reactor (ATR) rig has undergone intensive experimental and numerical characterisation by varying its motion over a range of parameters. The goal was to characterise the basic dynamics of the internal agitator with and without solid particles, using a laser displacement device, high-speed video and an ultrasonic velocity profiler (UVP) for three test cases ($f \approx 3 - 5$ Hz). The experimental data was compared to computational fluids dynamics (CFD) simulations of the *ATR* motion under the same operating parameters (frequency and displacement). There was very good agreement between simulation and experiment, in terms of agitator dynamics and fluids velocity field, demonstrating the numerical model can accurately predict the system: the velocity profiles and associated RMS through the reactor were determined, highlighting the complex coupling of the agitator and fluid, indicating areas of relatively low mixing in the centre and upper segments of the reactor. The CFD simulation was then used to calculate the power dissipation per unit volume, suggesting the 5 Hz case was the most effective for particle dispersion. Initial testing of the ATR with catalyst loading between 0.1 – 1 wt%, showed the solids did not cause measureable changes in reactor movement or fluid velocities. A phenomenological model – to be confirmed with numerical and experimental results – suggests that there is a critical frequency below where the power input is negligible. Above the critical agitation frequency, the power per unit volume varies as f^3a^2. Finally, the influence of turbulence on particle dispersion was correlated to direct numerical simulations (DNS) of particles suspended in a turbulence ‘bomb’, wherein turbulence levels correlating to those in the reactor are generated without any bulk fluid motion. In all simulations over a particle-size range of 5 – 500 μm, particles > 10 μm were observed to sediment to some degree, suggesting that there may be dispersion issues within the ATR for larger catalyst particles. However, there were also clear differences to particle transport in a DNS of a channel flow used for comparison, and as there was no direct experimental evidence for catalyst sedimentation, it may be inferred that the DNS did not fully capture the micro-scale hydrodynamics of the ATR's oscillatory motion

Yields and reaction kinetics of a model solids-catalysed reaction (oxidation of benzyl alcohol catalysed palladium-impregnated carbon particles) were determined in the batch-tests, while comparisons to the same reaction in the ATR at various frequencies are part of ongoing investigations, and will be reported in a re-issue of Deliverable D6.3.

TRL of PI Technology:

Technology Readiness Level (TRL) of the pilot-scale Coflore reactor at beginning of the study was estimated at ~5. TRL at the completion of the study, including knowledge gained form the full hydrodynamic and chemical characterisation of the reactor for solids catalysed reactions, as well as the scale up studies, is estimated to be 7. We are confident that the reactor will reach production.

8. List of Publications from WP6

8.1. Journal Publications

He Y., Bayly A. E., Hassanpour A. (2018) Coupling CFD-DEM with dynamic meshing: A new approach for fluid-structure interaction in particle-fluid flows. Powder Technology 325, 620-631. *[relates to Case Study 5]*

He Y., Bayly A. E., Hassanpour A., Muller F., Wu K., Yang D. (2018) A GPU-based coupled SPH-DEM method for particle-fluid flow with free surfaces, Powder Technology 338, 548-562. *[relates to Case Study 5]*

8.2. Conference Presentations

Boodhoo, K. V. K., Phan, A., Zivkovic, V., Eze, V., Mustaffar, A. (2018) Intensified-By-Design (IbD): Creating a Platform for Facilitating Process Intensification in Solids Handling Applications, AICHE 2018 Spring Meeting and 14th Global Congress on Process Safety, April 2018, Orlando, United States. *[relates to Case Study 4.1]*

Hunter, T. N. (2018) Use of acoustic backscatter systems to characterise concentrated dispersion. World Congress of Particle Technology, April 2018, Orlando, Florida, United States *[relates to Case Study 5]*

Mustaffar, A., Phan, A., and Boodhoo, K. V. K. (2018) Bi-Directional Thermal Control of Twin Screw Granulation Process Via a Specialised Annular Heat Pipe, AICHE 2018 Spring Meeting and 14th Global Congress on Process Safety, April 2018, Orlando, United States. *[relates to Case Study 4.1]*

Ohenoja, M., Ruusunen, M., Koistinen, A., Kaartinen, J., Paaso, J., Isokangas, A., Paavola, M. (2018) Towards Mineral Beneficiation Process Chain Intensification. IFAC MMM 2018, The 5th workshop on Mining, Mineral and Metal Processing. August 2018. *[relates to Case Study 3]*

Paavola, M., Okkonen, M. (2018) Optical measurements for flotation monitoring and diagnostics. NDT2018, The 57th Annual British Conference on Non-Destructive Testing. September 2018 (Accepted) *[relates to Case Study 3]*

8.3. Miscellaneous

On-line Measurements in Flotation Processes with Raman Spectroscopy and Camera-Based Methods. Paaso J., Seminar presentation in OMS R&D Centre opening seminar and Kick Off Ceremonies 22.5.2018 (Co-operation between Mining industry and University research and education), Oulu, Finland, 22.5.2018. *[relates to Case Study 3]*

Intensified by Design (IbD®). Paavola, M., Ohenoja, M., Koistinen, A., Ruusunen, M., Isokangas, A. Seminar presentation in Tekniikan Torstai –seminar series (in Finnish), Oulu, Finland, 5.4.2018, https://www.youtube.com/watch?v=mcXOXTFONbU. *[relates to Case Study 3]*

Monitoring and control in mining. Koistinen, A., Juuso, E., Seppälä, P., Paavola, M., Ohenoja, M. Poster presentation in FinnMateria 2016, Jyväskylä, Finland. 23.11.2016. *[relates to Case Study 3]*