Case Study 3: 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 1, 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.

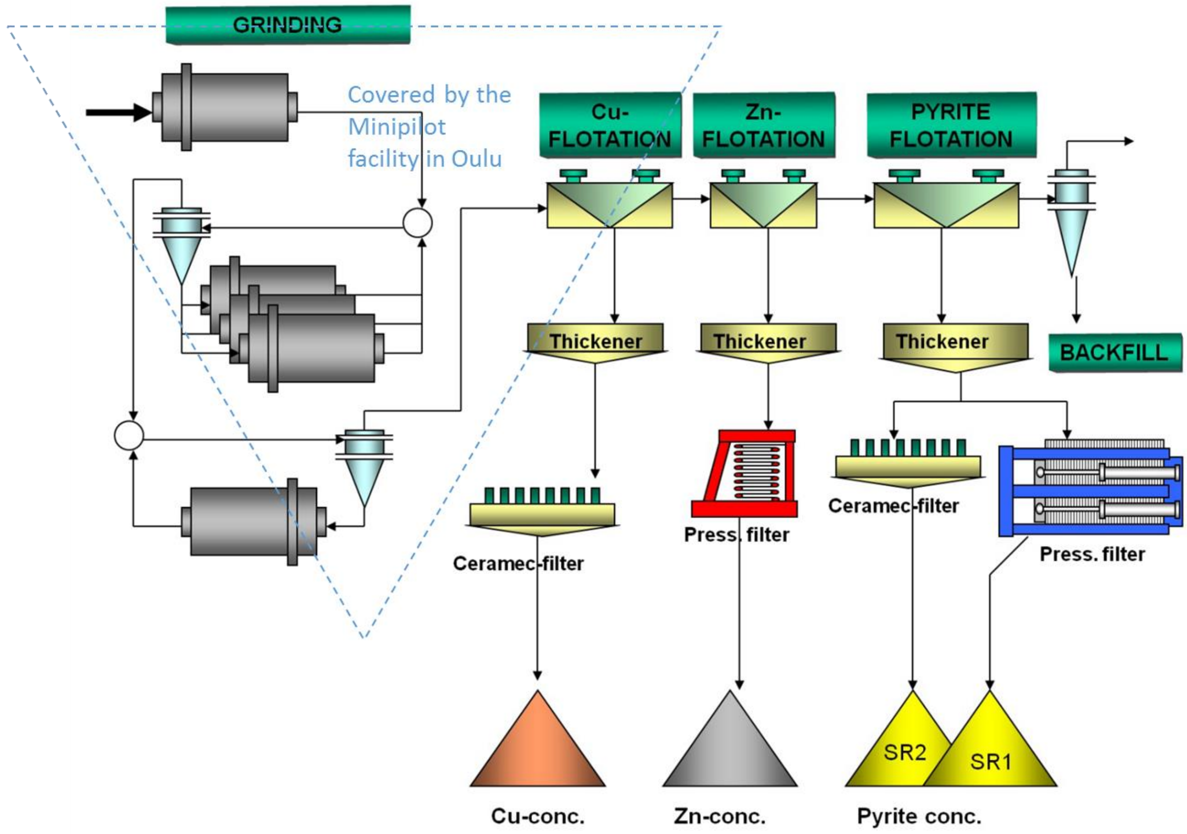

Figure 1. Process description of the Pyhäsalmi concentrator plant.

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 2, 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.

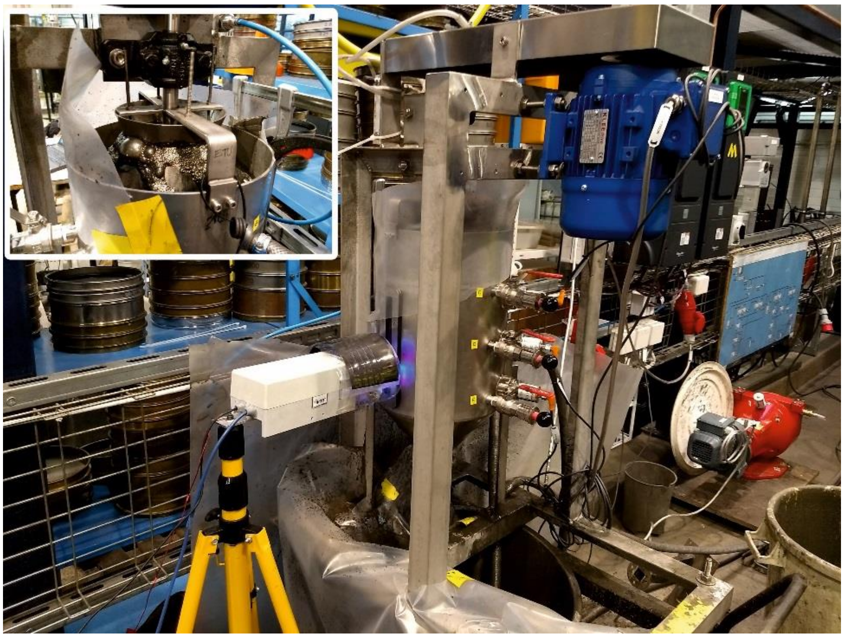

Figure 2. Coarse flotation device in minipilot-scale. On top, a copper concentrate is flowing from the top of the device. In the lower left corner, the particle tracking measurement device is also seen.

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 3. 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 4.

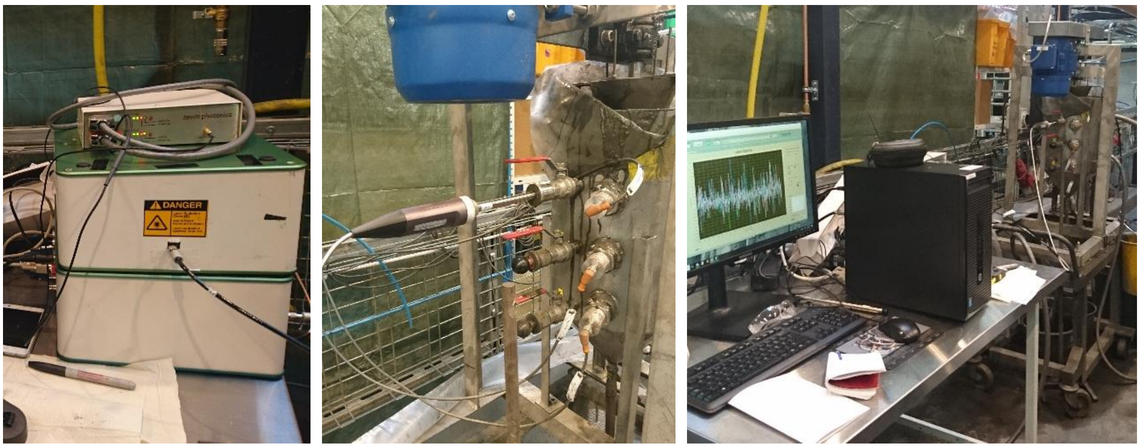

Figure 3. a) TimeGated Raman instrument M1, b) customized Marqmetrix Performance Ball Raman Probe connected to coarse flotation cell and c) TimeGated instrument PC connected to the M1 spectrometer next to the flotation cell.

Figure 4. 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 5, continuously updates the flowsheet model with the measured data from the real process.

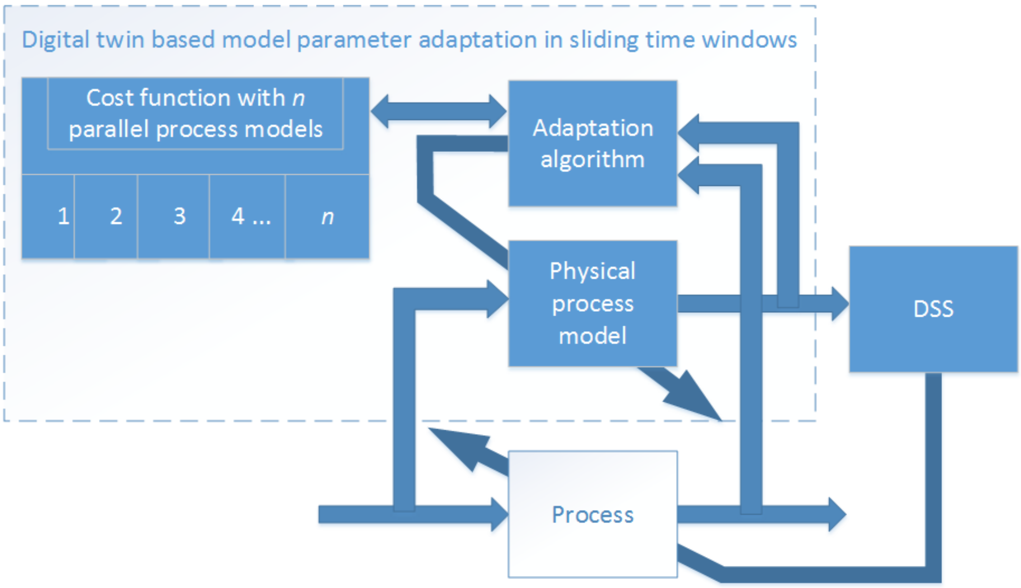

Figure 5. Resulted model parameter adaptation scheme**.**

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 after all formal ON/OFF trials have been completed. At the moment, the trials have indicated an 25% degradation in particle size standard deviation and an improved energy-efficiency of the grinding circuit with estimated annual energy savings of 75 MWh (0.5%). The results can be considered good taking into account the very high efficiency of the Pyhäsalmi mine.

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 dynamic mass balancing is in everyday use in Pyhäsalmi concentrator plant.
- It is estimated that the advanced grinding circuit control alone can result as 75 MWh annual energy savings in Pyhäsalmi concentrator plant.
- 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 at the minipilot scale didn't support the full-scale implementation with the studied ore.
- The quantified process intensification impact can only be seen after a longer period of operation than that available during the project timeline.

- 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.

Overall, PI is achieved in terms of less energy input in grinding, higher plant throughput and increased recovery. Mineral beneficiation is an energy-intensive industry on a global scale. Especially particle size reduction by grinding consumes about 4% of the world's energy. This case study explores an approach to avoid excess use and loss of energy in mineral beneficiation processes. The demonstrations focuses on sulfidic ore types, but the methods for redesigning of process flows, capacities, and product particle size are applicable in any base metals, ferrous, or industrial mineral beneficiation plants.

Development of the coarse flotation machine and improved process chain management will allow the use of substantially less energy in the upstream grinding process which in turn has environmental benefits (grinding takes 30-40% of the whole plant's energy). Improvements in efficiency by 10-30% are expected. Total recovery of valuable metals should be increased by 1-3%.

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).

1. 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.

Freeman Technology, Tewkesbury, 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.

For this case study two intensifications had been performed, the intensification of the L-thyroxine drug manufacture process at Sanofi facilities, detailed in earlier reports, by performing only one standard high-speed granulation per batch, by concentrating four times the premix of the active ingredients. This method would reduce the number of granulations per batch, and in consequence, reduce the process time per batch, and the Twin Screw Granulator process intensification. Both processes have demonstrated an environmental improvement, mainly due operation input/output flow demands.

The efficacy of continuous granulation for this drug formula was examined 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 6): 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.

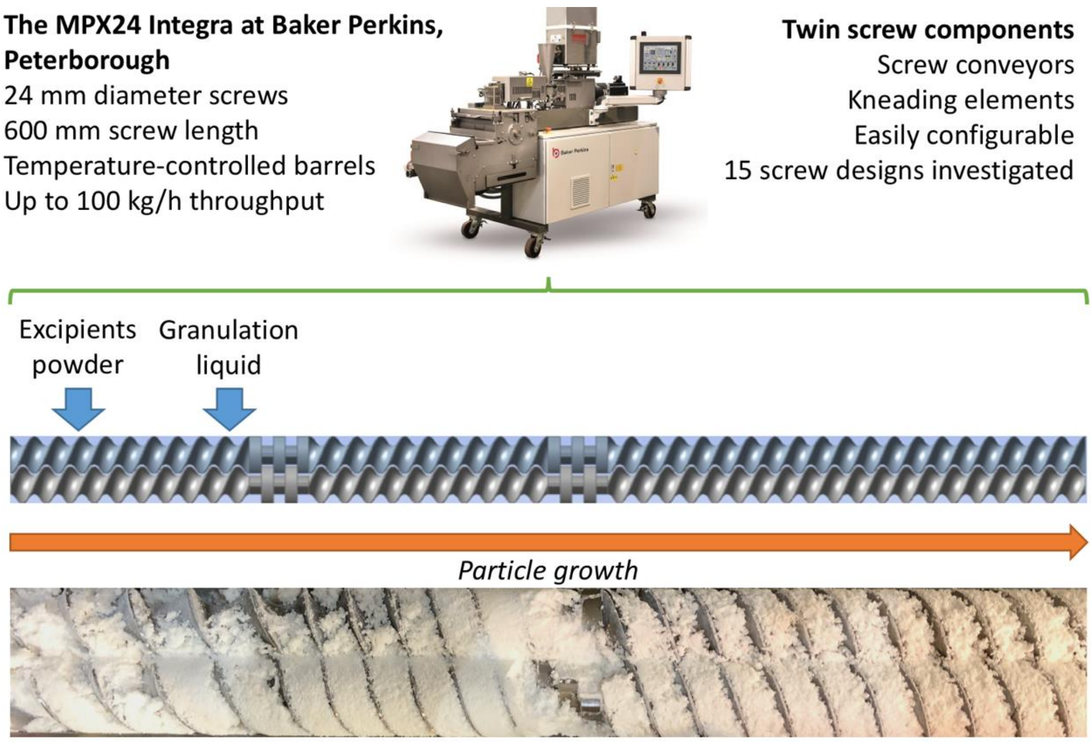

Figure 6. The twin screw granulator. The twin screw design shown was only a representative design out of fifteen designs investigated.

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 7, 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.

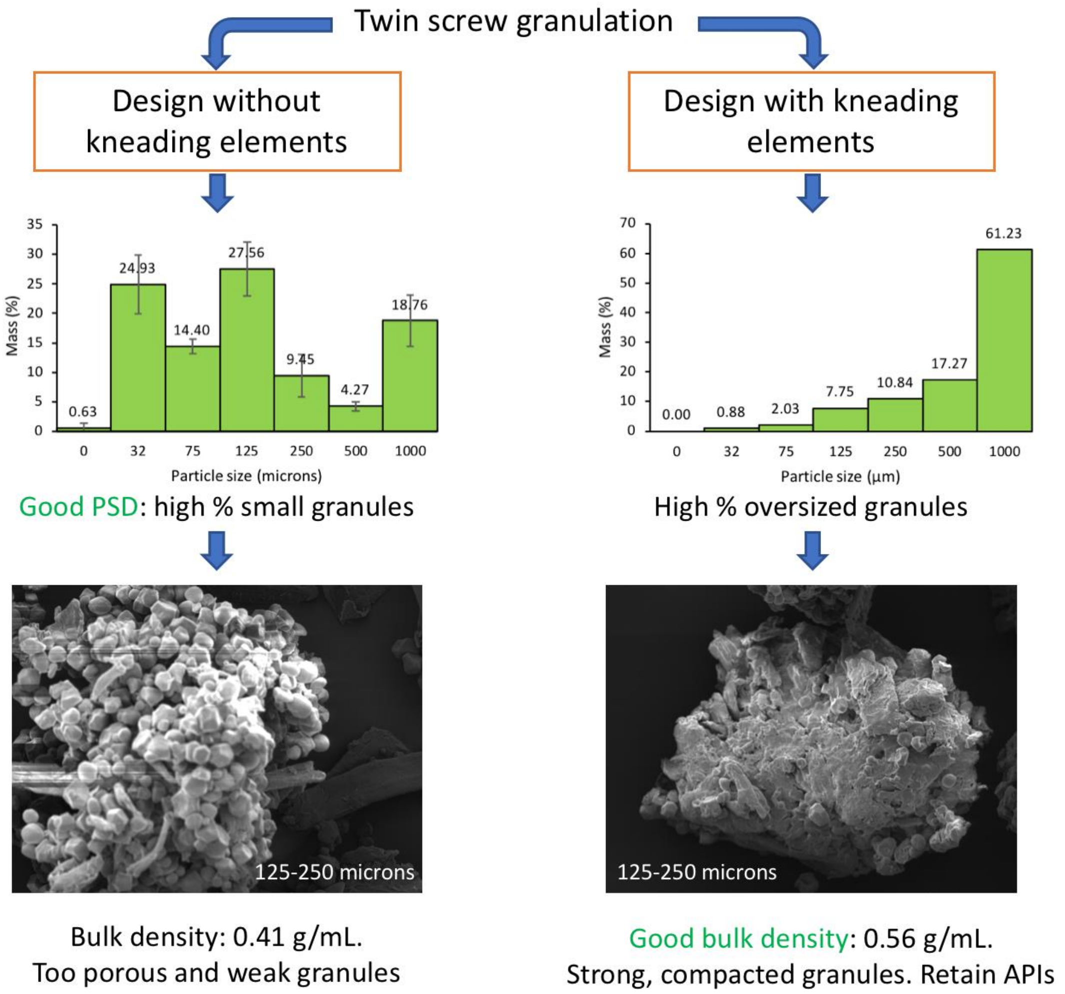

Figure 7. The effect of screw designs (e.g. with/without kneading elements) on granule properties was found to be substantial.

Final conclusions from the Case Study:

The analysis of the intensification of the L-thyroxine drug manufacture process at Sanofi facilities, has allowed an overall reduction of the impacts in all impact categories (between 4% and 70% of reduction). From the stages identified with the highest reduction potential in the conventional process (raw materials and operation), the operation phase is the one which has contributed mainly to the impact reductions, due to the improvements made; these improvements have achieved an impact reduction of approximately 75% in each impact category, including a 75% reduction in energy costs.

The tests on the twin screw granulator have demonstrated its viability 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.

Remarks on energy efficiency of the TSG process

The energy consumption for the conventional process of L-thyroxine production in Sanofi facility is 13.86 kWh/kg. This figure includes all process unit operations, namely: mixing, granulation, drying, tableting, packaging, etc. For the intensified processing carried out in Sanofi, the figure was reduced to 3.47 kWh/kg, a reduction of 75% from the conventional process.

Regarding the twin screw granulator, the total reduction is very similar to the previous intensification, since the intensification of both processes are related to the operation phase, minimising impacts from the same inputs and outputs and so on the environmental benefits of whole process are very alike. Taking this into consideration, the reduction achieved from the twin screw granulator process intensification goes from 4% to 70% depending on the impact category. Focusing on the operation phase, the one that has been intensified, the reduction in all the impact categories is above the 70%, while for the equipment construction phase the benefits are defined between the 62% and the 93% depending on the environmental indicator.

As a general conclusion, it can be stated that the intensification actions carried out by Sanofi and UNEW in the granulation process has improved the sustainability profile of the overall manufacturing process, both from an environmental and economic perspectives.

TRL of PI Technology:

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