Revolutionary Methods Require New Approaches

Process Intensification Methods: from Lab to Industry

Process Intensification is a relatively new approach to boost a plant's efficiency: It combines the optimisation of capital, energy, environmental and safety benefits with a radical reduction in plant-size. But the application of such revolutionary techniques has to overcome obstacles. Elaborate design and specialised equipment are needed to deal with unconventional constrains.

SÉBASTIEN ELGUE AND MICHAEL SEIPEL

Picture: By courtesy of INPT and Mepi

Pilot scale demonstration — perhaps the most important step in process intensification at the MEPI

In the context of fine or pharmaceutical chemistry, Process Intensification presents a set of radically innovative principles in process and equipment design (micro process engineering, flow chemistry, micro reactor technology, ...), which can bring significant benefits in terms of efficiency, capital and operating expenses, quality, wastes, safety, and so on.

Introducing this new method technologies requires significant investment in an environment where payback times need to be short. Large investments in current technology and limited experience hinder the introduction of Process Intensification. Managing the technical and financial risks requires new scale-up approaches and piloting facilities.

Many barriers to implementation of Process Intensification start with the lack of a suitable pilot & demonstration plant. Furthermore, high technical and financial risks exist in the development of an industrial prototype and the first implementation (retrofitting) into existing production lines.

Developing the Right Tools

To overcome this barrier, the need for facilities where Process Intensification methods can be developed, piloted and demonstrated on a semi-industrial scale is eminent.

To develop such an approach, the National Polytechnic Institute INPT of the Toulouse University has started the creation of MEPI (Maison Européenne des Procédés Innovants, French for "European House of Process Innovations"), a demonstration facility for Process Intensification technologies. In this context, the association between INPT and MEPI aims at matching strategic and applied research, piloting and industrialization and consulting and knowledge dissemination.

With regards to Process Intensification, the choice of the suitable equipment for a specific application is not obvious. Therefore, INPT has developed methodologies and tools for equipments choice, based on its knowledge of each technology. The objective of

Mr. Elgue works for the Maison Européenne des Procédés Innovants. Michael Seipel works for LAUDA Dr. Wobser GmbH & Co KG. Contact LAUDA: Phone +49 (0) 93 43 / 5 03 - 27 3

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such an approach is to propose the optimal technology for the user, as each equipment presents its own advantages and drawbacks.

The recipe of a successful intensification lies not only in optimal design and/or control of the process but also in the process environment. In most cases, the key-points of process environment consist of dosing lines and pump technology, of thermal control loops and the control system. Regarding the process environment, INPT and MEPI have developed significant skills and know-how, based on collaborative relationships with equipment suppliers.

Reactor Design is Result of Studies

A recent study emphasizes the importance of this strategy: It deals with the transposition from batch to continuous and process intensification of an API production, including reactions and post treatment.

The synthesis of this product involves two main steps: an initial formation at an optimal temperature of 30 °C and a racemisation at a temperature level above 100 °C.

To obtain the desired product quality it is of major importance to control the application's temperature thoroughly. For this prupose a new kind of heat exchange reactor made of silicon carbide, offering both high thermal conductivity and corrosion resistance, has been developed in a partnership between Boostec (of Mersen Group) and INPT.

The setup of this new reactor type is based on many elaborate lab studies. The reactor design deals with geometrical considerations like dimension and shape of channels but also with practical considerations like the application of seals and connections.

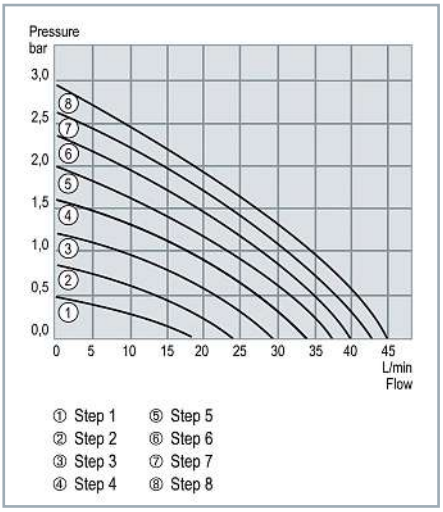
Regarding the properties of the reaction, special care was taken of the utility fluid loop to optimise the temperature control. The stainless steel utility plates are specially designed, including fins. The selected steel increases the plates' thermal capacitance and so the passive safety of the process.

Comply Unconventional Constraints

To obtain the best performances and comply with the "unconventional constraints" of the process, specific features are required regarding the heating/cooling device. To meet this challenge, a process thermostat from Lauda is used. Lauda's Integral XT 150 provides a strong heating and cooling capacity to cope with rapid temperature changes. The combination of the small diameters of the reactor connections and the small channels inside the device calls for a strong pump with high gauge pressure. Therefore the thermostat is equipped with a pump that delivers a maximum pressure of 2.9 bar and a flow rate up to 45 l/min. The eight levels of the vario pump enable the adaption of the pressure and flow supplied to the reactor.

The resultant reactor offers at pilot scale enhanced thermal performances with a heat exchange capacity (UA/V) of 20,000 kWm^2/m^3.

Following the developed methodology, the process has been thoroughly characterised in terms of hydrodynamics, mixing, heat and mass transfer etc., according to benchmark procedures developed by INPT specialists.



Characteristics of the vario pump of the Integral XT thermostat. The pump can be set in eight individual levels to adapt to a specific process, with a maximum pressure of 2.9 bar and a flow rate of 45 l/min.

From Lab to Industry Application

To speed up the transposition to continuous processes and improve the process control, an in-line analysis based on Raman spectroscopy has been set up. Satisfactory performances during benchmark steps allow the equipment to be transferred to MEPI, where an industrial demonstration at pilot scale is prepared. This final demonstration step allows to perform optimal control of the process and to prepare the installation on the customer production site.

Eventually, the full continuous and intensified process including reaction and post-treatment methods (like precipitation, filtration, drying) has been validated during production campaigns on end user site at a pilot scale, leading to an API production of seven tons per year. ●