Project no. NMP2-CT-2005-011816

IMPULSE

Integrated Multiscale Process Units with Locally Structured Elements

Integrated Project (IP)

Thematic Priority 3: Nanotechnologies and nano-sciences, knowledge-based multifunctional materials and new production processes and devices (NMP)

Publishable Final Activity Report

Period Covered: from 1 February 2005 to 31 January 2009

Date of preparation: 10 November 2009

Start date of project: 1 February 2005

Duration: 48 months

Project Coordinator: Michael Matlosz

Revision 3.0

Project coordination organisation name: Centre National de la Recherche Scientifique (CNRS)

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1. IMPULSE : the breakthrough of structured multiscale design

The goal of the IMPULSE flagship integrated project in the 6th Framework Programme was a clear breakthrough for radical long-term transformation of the chemical industries in Europe. This goal was to be achieved through locally targeted integration of innovative, intensified process equipment (such as microstructured reactors, compact heat exchangers, thin-film devices and other micro- and/or meso-structured components) for enhanced whole-process performance, thereby contributing to supply-chain sustainability and competitiveness.

The IMPULSE approach, entitled "structured multiscale design", represents a paradigm shift in the concepts and methods of chemical and process engineering : rather than adapting operating conditions to equipment limitations, structured multiscale design adapts the equipment structure and process architecture to enable achievement of the most desirable operating conditions for a given physico-chemical transformation. Attaining widespread implementation of this new approach requires clear proof of principle case studies, successful demonstration activities in several industrial sectors and development and dissemination of generic methodological concepts and tools. This is precisely what IMPULSE has done.

The success of the IMPULSE project is the achievement of nine specific case studies, resulting in five industrial pilot demonstration units in the industrial sectors of pharmaceutical products, specialty chemicals and consumer goods, as well as the development of appropriate design methods, tools and training material.

The demonstration units provided evidence for reliable, cost-efficient and cost-effective integration of small-scale, high-performance structured equipment into large-scale production. The illustration of continuous hydrogenation for pharmaceutical synthesis in the GlaxoSmithKline demonstration unit in Tonbridge (UK) addressed multiscale design benefits for eco-efficiency and safety. The illustration of highly exothermic liquid-liquid alkylation for ionic-liquid production in the Solvent Innovation demonstration unit constructed at RWTH-Aachen (D) provided evidence for the interest of targeted continuous heat-transfer and mixing devices, as well as the safety advantages of reduced inventory for toxic reactants. The illustration of miniaturized surfactant production with the Procter and Gamble demonstration unit in Brussels (B) opened the path to eco-efficient use of decentralised, delocalised production for the fabrication of high-tonnage consumer products, and the two additional Procter and Gamble microfluidic demonstration units in Brussels for continuous microencapsulation and for continuous emulsion vesicle formation illustrated the scale-up capability of the IMPULSE structured multiscale approach for product formulation technologies.

The visibility of these five successful demonstrators, associated with the IMPULSE project label, is an important factor in the promotion of innovative multiscale technologies as a significant contribution to sustainable and competitive chemical process industries in Europe.

In addition to the demonstration activities and case studies, the generic methodological research in IMPULSE achieved major breakthroughs in the development of the concepts required for structured multiscale design and in the translation of those concepts into forms suitable for training activities targeted specifically to industrial audiences. The IMPULSE training material developed for this purpose was tested within the organizations of several IMPULSE industrial partners, and complementary training material for academic audiences was developed as well. Beyond the IMPULSE consortium partners, the strong interest expressed by more than a dozen companies outside of the IMPULSE consortium, including equipment manufacturers, in the activities of the IMPULSE User Group and in the creation of the IMPULSE equipment database, offered additional visibility for extensive uptake of the new approaches throughout European industry. Following project completion, the legacy management arrangements for IMPULSE, including generalised publication of the IMPULSE "Big Book", summarising the essential features of the methodological developments, the creation of a specific IMPULSE web portal for information exchange, and the integration of IMPULSE results in the foundations of at least three new projects in the 7th Framework Programme, provide a strong basis for continued widespread dissemination of the IMPULSE approach to a large audience in both the industrial and academic communities.

2. The IMPULSE Project

2.1 Background

The European chemical industry is currently the largest in the world, with over 30 % of total annual world-wide production, representing more than 12 % of total EU gross added value in the manufacturing industries and more than 2,7 million jobs. This leading position is nevertheless seriously threatened by substantial growth and investment from major competitors in North America, Japan, India and China. Furthermore, the new challenges generated by the quest for truly sustainable development create an urgent need for innovative process solutions for safer, cleaner and more economically and energetically efficient production facilities. Improved competitiveness and sustainable development in the chemical sector require radical improvement therefore not only in the discovery and use of new molecules and chemicals, but also in the chemical processes that produce them.

As international leader in the areas of process intensification and microreaction technology, the European chemical industry and research communities are in an excellent position to respond to these global challenges through innovative production technologies based on collaborative industrial-academic research on the European level.

2.2 Project Objectives and Expected End Results, Use and Impact

The overall objective of IMPULSE was the effective and targeted integration of innovative process equipment such as microreactors, compact heat exchangers, thin-film devices and other micro and/or meso-structured components to attain radical performance enhancement for whole process systems in chemical production.

The approach developed in IMPULSE, known as structured multiscale design, represents a new design methodology for chemical process engineering to attain reliable, efficient and cost-effective integration of small-scale, high-performance structured equipment and devices into large-scale production systems.

The goals of the IMPULSE project were the following:

- proof of principle of IMPULSE approaches in three supply-chain sectors (pharmaceutical products, specialty chemicals and consumer goods)
- validated business cases for selected applications
- “teachable” generic design methodology and optimisation techniques
- evaluation of multiscale design benefits for eco-efficiency, safety and sustainability

Pilot demonstrator units from specific application areas in supply-chain sectors, were planned and developed from the RTD results by the industrial partners, and wide-spread dissemination of the IMPULSE approach was ensured through the development of training material, conference presentations, newsletters and a user group.

2.3 Description of Work

To provide proof-of-principal for the IMPULSE approach and build the necessary foundations for the design methodology as well as the future demonstration units, specific sectorial RTD tasks were performed for the following nine targeted applications:

A “pharmaceutical products” subproject (SP1) included the following tasks:

- T1.1: continuous hydrogenation
- T1.2: continuous solids handling
- T1.3: integrated primary and secondary manufacturing

A second “specialty chemicals” subproject (SP2) included the following tasks

- T2.1: liquid-liquid alkylation
- T2.2: miniemulsion polymerisation

- T2.3: electrochemical alkoxylation

A third specific sectorial subproject on “consumer goods” included the following work

- T3.1: sulfonation and sulfation
- T3.2: targeted encapsulation
- T3.3: controlled emulsification

The principal process technology objectives for these specific application case studies were the following:

- replacement of batch processes by steady-state continuous flow systems,
- modular processes for variable throughput and mass customization
- integration/connection of innovative equipment for retrofit into existing plant
- miniaturization of process systems for distributed, delocalized production

Cross-sectorial RTD in a specific workpackage (WPB) was dedicated to over-arching methodological research covering the following generic issues:

- TB.1: whole process design
- TB.2: laboratory protocols
- TB.3: equipment design
- TB.4: business and SHE
- TB.5: information exchange and analysis

2.4 Partners involved in the contract

The table below presents a list of the partners involved in the project

Participant name	Participant short name	Country
Centre National de la Recherche Scientifique	CNRS	F
ARTTIC	ARTTIC	F
Britest Limited	BRITEST	UK
DECHEMA Gesellschaft für chemische Technik und Biotechnologie, e.V.	DECHEMA	D
Degussa	DEGUSSA	D
URV : Escola Tecnica Superior d’Enginyeria Quimica de Tarragona	URV-ETSEQ	E
Forschungszentrum Karlsruhe	FZK	D
GlaxoSmithKline	GSK	UK
Institute of Chemical Process Fundamentals of the Czech Academy of Sciences	ICPF	CZ
Institute of Chemical Technology Prague	ICTP	CZ
Institut für Mikrotechnik Mainz	IMM	D
Institut National de l’Environnement Industriel et des Risques	INERIS	F
Institut National Polytechnique de Lorraine	INPL	F
Procter and Gamble International Operations	PGIO	CH
Rheinisch Westfälische Technische Hochschule Aachen	RWTH	D
Siemens AG	SIEMENS	D
Solvent Innovation GmbH	SOLVENT	D
Nederlandse Organisatgie voor Toegepast-Natuurwetenschappe Lijk Onderzoek	TNO	NL
University of Manchester	UNIMAN	UIK
Warsaw University of Technology	WUT	PL

The project coordination was ensured by the CNRS (Centre National de la Recherche Scientifique) in France

IMPULSE Project Direction was ensured by Professor Michael Matlosz.

3. A yearly account of the IMPULSE project RTD results

The RTD results obtained by the IMPULSE project are summarised in the sections below on a yearly basis.

3.1 Year 1 (from February 2005 to January 2006)

Work performed and major RTD results achieved at month M12 are summarised below.

3.1.1 Sector-specific application RTD

3.1.1.1 SP1 – Pharmaceutical Products

A selection and validation of target hydrogenation reactions was carried out (including hydrogenation of substituted nitro-aromatics and hydro-deprotection), and clearance with respect to basic compatibility of the targeted processes with structured reactors.

Initial qualitative models (for filtration and dewatering) were drafted and circulated, and a survey of equipment design and selection critera for solids processing was launched.

A value stream process map created for a specific tablet dose form covering both primary and secondary operations, and a collation of specific business drivers for the process was performed.

3.1.1.2 SP2 – Specialty Chemicals

Characterization of reaction kinetics for alkylation of alkylsubstituted imidazolium derivatives with alkyl sulfates was performed, and the design and construction of an original continuous-flow laboratory prototype was completed.

Laboratory trials and modeling of monomer conversion kinetics with monomer/initiator/hydrophobe mixtures were completed, and initial design and fabrication of two laboratory-scale microstructured plate reactors with integrated heat exchanger structures was carried out.

A study of electrochemical reaction kinetics and selection of electrode material, electrolyte and operating conditions for the methoxylation of 1,4 methoxybenzene was undertaken, and the construction of a laboratory-scale prototype was completed.

3.1.1.3 SP3 – Consumer Goods

Process design work began, including strategy for integration into the process chain available, and initial experimental work on materials and devices, including corrosion tests.

The design principle for adaptation of a segmented flow tubular reactor (SFTR) was completed, and initial tests and encapsulation analysis and comparison of two different encapsulation chemistries (interfacial polymerization and phase-inversion polymerisation) was undertaken.

Emulsification performance and limits of microstructured devices for selected industrial application were evaluated, and simulation studies in view of design adaptations for structured devices were undertaken.

3.1.2 Cross-sectorial methodological RTD

The specification of the scope of SHE (Safety, Health and Environment), reliability and layout issues were completed, and initial reports on simulation of whole-process performance were prepared.

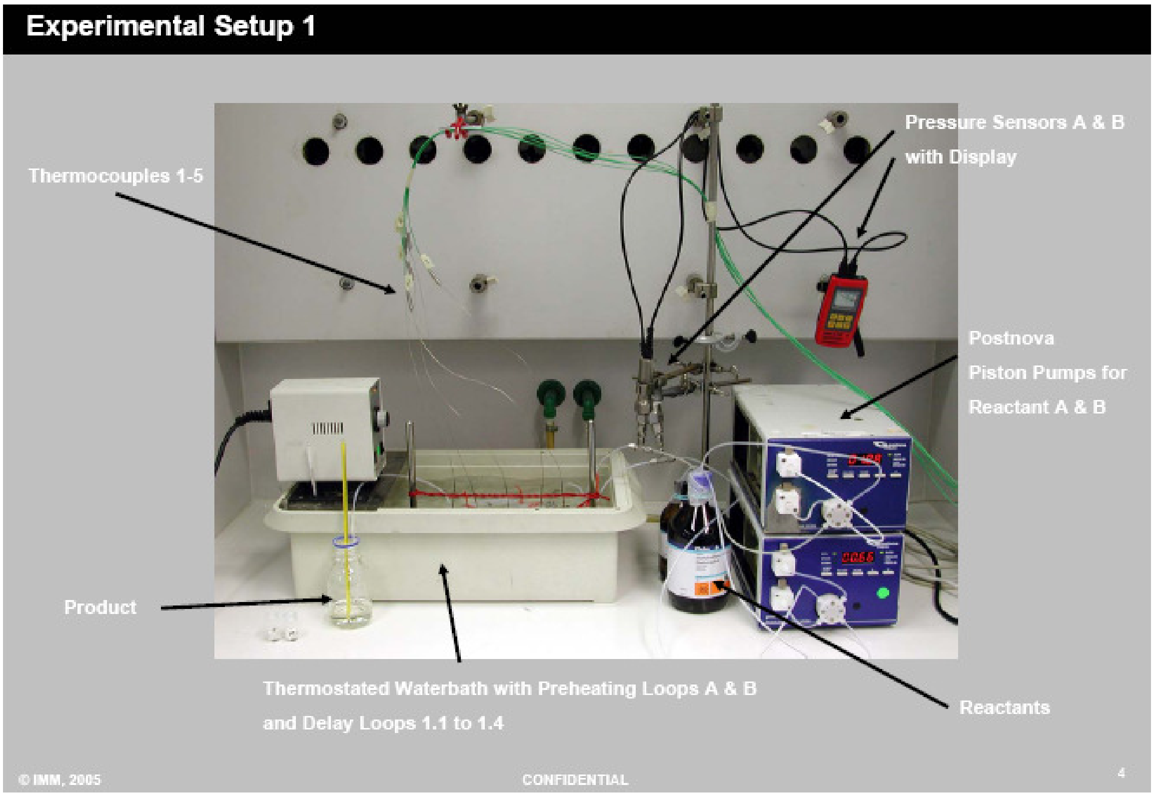
A critical assessment of existing methods for structured component characterization was undertaken, and a collection of typical structured element geometries were prepared.

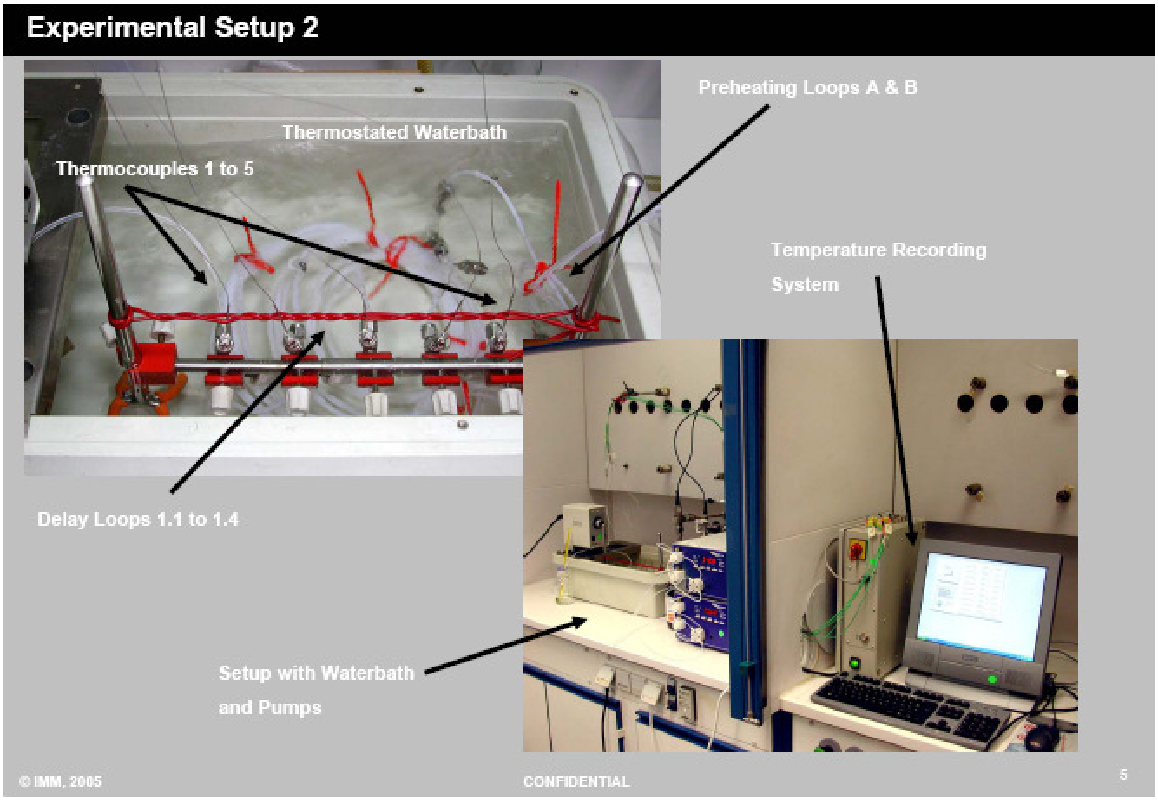
Theoretical studies in support of equipment design and scale-up were performed, and a proforma for characterizing equipment to allow matching to process requirements was completed.

Reports on proposals for environmental, economic and safety indicators were completed, and a critical evaluation of the three separate indicators was performed.

3.1.3 Experimental set-ups

Experimental set-ups of the work carried out in year 1 are shown in the pictures below:





3.1.4 Publishable results and dissemination

Among the publishable results in open fora the following two articles in international journals were highlights of year 1:

- Bayer, T., et al., "IMPULSE – A New Approach to Process Design," Chemical Engineering and Technology 28 (4), 431-438 (2005).
- M. Matlosz, "Mikroverfahrenstechnik : Neue Herausforderungen für die Prozessintensivierung," Chemie Ingenieur Technik 77 (9), 1393-1398 (2005).

Two major conference presentations represent also significant highlights for year 1:

- Matlosz, M., "Multiscale Design : Methods and Tools for the Sustainable Chemical Industry of the Future," 7th World Congress on Chemical Engineering, (Glasgow, UK, August 2005).
- Matlosz, M., "Multiscale Design : Methods and Tools for the Sustainable Chemical Industry of the Future," 4th International Workshop on Micro Chemical Plants (Kyoto, Japan, January 2006)

Two deliverables from the cross-sectorial work were made public, in particular as a basis for discussions in the IMPULSE User Group and for exploitation by equipment manufacturers and in the development of testing methods and standards.

The first IMPULSE Newsletter was released externally in October 2005.

3.2 Year 2 (from February 2006 to January 2007)

Work performed and major RTD results achieved at month M24 are summarised below.

3.2.1 Sector-specific application RTD

3.2.1.1 SP1 – Pharmaceutical Products

A first continuous hydrogenation reactor prototype was designed, built and tested, and a preliminary analysis of safety and eco-efficiency was performed. Investigation on the potential feasibility of a possible demonstration activity began.

A single model solid-liquid system was identified – namely sugar in a water-IMS solvent mixture. The system was analysed and characterised for filtration. A gap analysis of capabilities of equipment for isolation was performed and strategies for solids processing were evaluated.

Based on the value stream process map created in year 1 for a specific tablet dose, initial key performance drivers and success criteria for integration were defined. In addition, in order to identify strengths and weaknesses of the supply chain and unit operations, user requirements for simulation were identified and the results communicated to WPB, the cross-sectorial methodological work-package.

3.2.1.2 SP2 – Specialty Chemicals

Three reactor concepts for improved synthesis of the target product were evaluated, and a first generation of general design rules for the alkylation reaction to a liquid product were identified. Given the promising results, a possible demonstration activity concerning this application was considered (in place of the initially planned demonstrator in T2.2).

Various reactor concepts were developed and evaluated by the partners and various polymer surface materials were tested to avoid polymer sticking. Due to the need for additional work to adapt the reactor materials to avoid sticking, the initially proposed demonstrator on miniemulsion polymerisation (T2.2) activity was cancelled.

Experimental studies of alkoxylation in the laboratory prototype were undertaken to evaluate the influence of reactor segmentation on process performance. In addition, tests were performed for identification of suitable ionic liquid solvents for electrochemical methoxylation.

3.2.1.3 SP3 – Consumer Goods

An experimental microstructured device setup for SO2 oxidation was designed and constructed. Feasibility studies of both liquid/liquid and gas/liquid oxidation with both vanadium oxide and platinum catalysts were undertaken. In addition, the neutralization reaction for surfactant production in mini-structured reactor devices was investigated.

Simulation and experimental investigation of phase inversion precipitation for the formation of capsules was performed. Perfume-containing capsules were produced in batch mode and initial preparatory work was undertaken for trials in a microstructured continuous flow system.

Experimental and theoretical studies, including in situ imaging techniques and numerical simulation, were performed to evaluate emulsification performance in microstructured devices for industrial application. Testing of laboratory prototype devices was pursued, as well as preparation for future demonstration activity, with the definition of a flexible microreaction test unit at a Procter and Gamble site in Belgium.

3.2.2 Cross-sectorial methodological RTD

Development of whole process modelling and prototype software was undertaken for a hydrogenation process (based on T1.1), a sulfonation process (based on T3.1) and an encapsulation process (based on T3.2). In addition, in collaboration with TB.3, conceptual design and representation of multiscale processes using restricted data was explored to allow mapping of process concepts onto possible combinations of plant items.

Laboratory protocols and methodological issues were developed for determination of chemical kinetics using micro-structured elements as laboratory investigation tools. A time scale analysis of industrial processes with respect to coupled rate processes was developed and a protocol was established to determine the effect of channel geometry and construction material on the gas-liquid flow, holdup and interfacial area in microchannel systems.

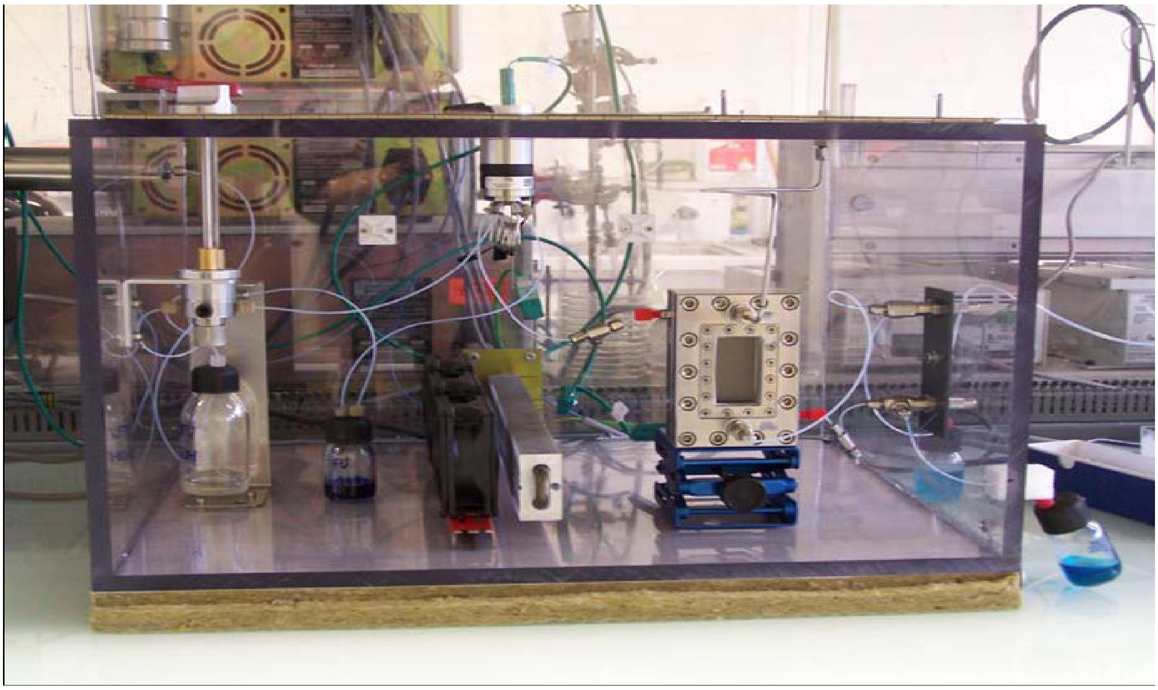
Development of a database of equipment characteristics for process development as well as methodology to assist preliminary equipment selection was pursued. In addition, RTD work on the adaptation of pressure and flow sensors based on the needs identified in subprojects SP1, SP2 and SP3 was undertaken.

Initial reports were completed on business and SHE (Safety, Health and Environment) work and delivered on the major issues and knowledge gaps to be examined for future methods and indicators for sustainable multiscale process design. Critical examination and assessment by the industrial partners of the proposed approaches and indicators was engaged.

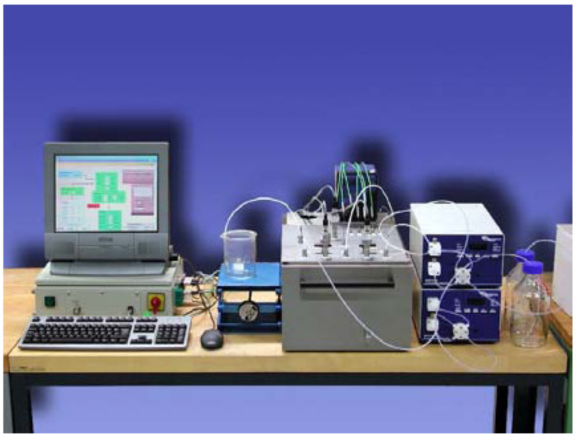
For information exchange and analysis, four facilitators were identified to lead information transfer activities and face-to-face meetings were undertaken with project partners in several SP tasks.

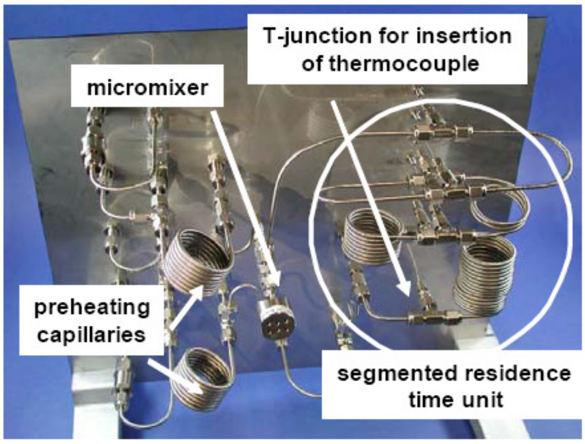
3.2.3 Experimental set-ups

Experimental set-ups of the work carried out in year 2 are shown in the pictures below.



Improved experimental setup for the synthesis of [EMIM][EtSO4]





3.2.4 Publishable results, dissemination and the first workshop

Among the publishable results in open fora, the following are year 2 highlights of articles in international journals:

- Loeb, P., et al., “Continuous microreactor rigs with capillary sections for organic synthesis: generic process flow sheets, practical experience and novel chemistry,” Chimica oggi – Chemistry Today 24 (2), 46-50 (2006),
- Minnich, C., et al., “Highly flexible fiber-optic ATR-IR probe for inline reaction monitoring,” Organic Process Research and Development 11 (1), 94-97 (2007).

The two major conference presentation highlights from year 2 are the following:

- Sharratt, P., "IMPULSE – the challenges in adopting a new processing paradigm," keynote lecture at the 17th CHISA International Conference of Chemical and Process Engineering (Prague, Czech Republic, August 2006).
- Falk, L., "Microstructured systems for industrial chemical production," keynote lecture at the 9th International Conference on Microreaction Technology (IMRET 9, Potsdam, Germany, 2006).

In addition, the first IMPULSE Scientific Workshop was held in July 2006 and the first IMPULSE User Group meeting organised in January 2007. The second and third IMPULSE Newsletters were released in May and December 2006. IMPULSE was present at ACHEMA 2006 and at numerous other forums and events.

3.3 Year 3 (from February 2007 to January 2008)

Work performed and major RTD results achieved at month M36 are summarised below.

3.3.1 Sector-specific application RTD

3.3.1.1 SP1 – Pharmaceutical Products

An appropriate fixed bed reactor was selected for the continuous hydrogenator to be used for demonstration activities, while research work proceeded on other advanced microstructured reactor systems. In parallel, a business case was made for the reduction of manufacturing costs.

The scope for design of equipment to carry out continuous filtration, continuous washing and continuous drying was defined.

The various tactics that should be employed for successful process integration of primary and secondary manufacturing were identified and assessed.

3.3.1.2 SP2 – Specialty Chemicals

The process design for the IMPULSE demonstrator plant for production of ionic liquids was completed. The new process included continuous microstructured equipment and was to be compared with a classical batch process. The process design package was the basis for basic and detailed engineering and included PFD (process flow diagrams), heat and mass balances, cost estimations and a complete description of the new process.

The root causes for the polymer blocking and particle growth which appear while running the miniemulsion polymerisation in a continuous mode were identified.

For electrochemical alkoxylation, a reactor design was developed that enables effective separation from the reaction mixture of gas evolved at the electrode surface of the microchannel cell.

3.3.1.3 SP3 – Consumer Goods

The design and construction of a third-generation micro falling film reactor (MFFR) suitable for demonstration purposes at an industrial scale was successful. The first trails performed confirmed the benefit of the new design. In parallel, a scaled-up version of the SO2-oxydation reactor to be implemented for the demonstration activities was developed.

Complete CFD modelling of the SIMM mixers was achieved. In addition several modified inlays and mixer elements were designed and built to make larger capsules. The lack of suitable in-line characterisation capability was addressed with an extensive development of designs for camera chambers. The understanding of the permeation of perfume components through polymer walls was further developed.

Various reactor types (caterpillar, V-type…) were investigated and the samples produced were analysed and compared. A scaled-up version of the caterpillar mixer for later use in the demonstration activities was designed and built. A new method and a new setup to be able to monitor the quality of mixing processes at different axial positions in the reactor using NMR techniques were developed.

3.3.2 Cross-sectorial methodological RTD

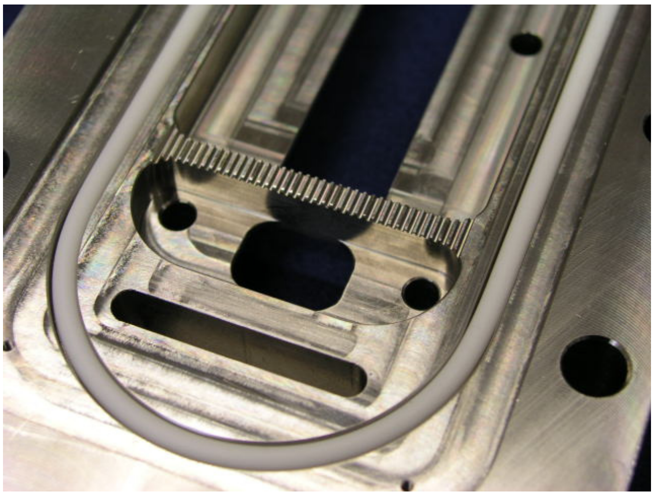
A draft of the whole process multiscale design methodology was released.

A first version of a design methodology of multiscale network structures was proposed, enabling linkage between micro, meso and macro elements. Enhanced performance of structured devices through systematic geometrical design, rather than the ad-hoc approach generally used, is now possible and should allow further improvements of device performance beyond current levels.

The initial equipment selection tool was finalized for rapidly choosing equipment suitable for carrying out a particular process task, as part of the design methodology for multiscale structured processes being developed in WPB. The tool uses a database of equipment characteristics developed within task TB.3.

A report with guidance on “how” to apply the European directives on pressure vessels and explosive atmospheres when processes are intensified with structured multiscale equipment was written. In parallel, a second report summarizing the present knowledge in decision making involving process intensification with multiscale equipment was also prepared and submitted.

The information exchanged during months 13 to 24 was analysed, and areas where further interaction and analysis would be useful were identified. Four workshops concerning these areas of generic interest were organised between key people from the generic work programmes (WPB and WPC) and the SP projects.

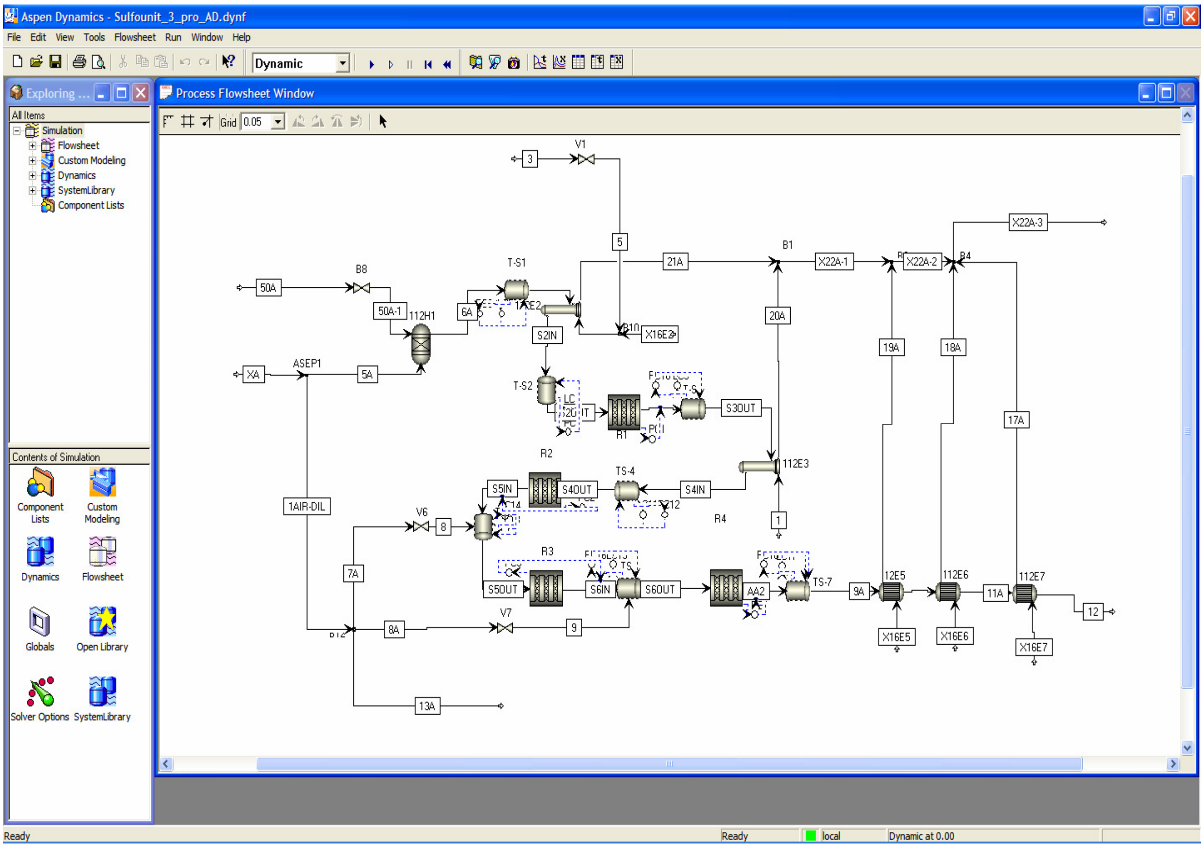


3.3.3 Experimental set-ups

A detail of the dosing system of newly developed falling film reactor in task T3.1 related to the work carried out in year 3 is shown in the picture on the left.

The gas enters through one or two holes and is led along the channels via microstructures, ensuring equal gas flow over the width of the channel. Liquid enters through the long hole below and is led along the reaction foil.

The dynamic model of sulfonation unit in ASPEN Dynamics environment is shown in the picture below



3.3.4 Publishable results, dissemination and the second workshop

Among the publishable results in open fora, the following are year 3 highlights for articles in international journals:

- Torras, C, Pitol, L, Garcia-Valls, R., “Two methods for morphological characterization of internal microcapsule structures,” Journal of Membrane Science, 305 (1-2), 1-4 (2007).
- Loeb, P., Hessel, V., Hensel, A., Simoncelli, A., “Micromixer based liquid/liquid-dispersion in the context of consumer goods production with focus on surfactant vesicle formation”, Chimica Oggi / Chemistry Today, 25 (3), 26-29 (2007).

The two major conference presentations highlights from year 3 are the following:

- Minnich, C., et al., “Redefined and extended investigation of microreactor based ionic liquid synthesis for further process intensification.”, European Congress of Chemical Engineering, Copenhagen, 16-21 September 2007.
- Davison S., Double J., Gourlay B and Sharratt P.N.; “Appropriate use of microreactors in pharmaceutical processes”, ISPE Paris Conference, 16th -17th April 2007.

In addition, the second IMPULSE Scientific Workshop was held in June 2007 and the second IMPULSE User Group meeting organised in January 2008. The fourth and fifth IMPULSE Newsletters were released in September 2007 and January 2008.

3.4 Year 4 (from February 2008 to January 2009)

Work performed and major RTD results achieved at month M48 are summarised here below.

3.4.1 Sector-specific application RTD

3.4.1.1 SP1 – Pharmaceutical Products

Work was carried out on developing/building the laboratory reactor prototypes and comparing the performance of the reactors on their targeted reaction. The laboratory work on the fixed bed reactor confirmed the design of the demonstration reactor, in particular the requirement to develop a duty/stand-by capability to manage catalyst deactivation. The relevant demonstration experiments were designed and performed in the reactor. From the lab and pilot scale results, an introductory methodology was developed for whole process design, reactor design, process scale-up, and operational rules for industrial manufacture. Generic learnings were captured regarding the limitations of structured elements for multiphase reactions.

For continuous solids handling, the working team selected and defined the means to prepare solid suspension systems for the testing of equipment capabilities. Reviews of the capabilities of the equipment that is currently available for solids processing and carrying out a gap analysis, so as to be able to design devices to fill capability gaps were carried out.

For integrated primary and secondary manufacturing work was carried out to characterise the nasal suspension process and equipment for existing non-integrated primary and secondary manufacturing unit operations. Additional characterisation work was done following the identification of unit operations to integrate the batch process and development of a conceptual continuous process. A core part of the work was on the development of a supply chain simulation model. This model enabled risk analysis and eco-efficiency evaluation of the existing manufacturing processes and the comparison of the new conceptual designs. The end result was the identification of technology required to integrate Primary and Secondary manufacture.

3.4.1.2 SP2 – Specialty Chemicals

Implementation of a newly designed continuous-flow lab prototype and the evaluation of the extension of the continuous–flow process to other highly exothermic liquid/liquid systems were carried out. Based on the knowledge obtained from the testing of the existing and the new reactor, the implementation of the highly exothermic L/L-reactions in a continuously operated structured reactor on bench or pilot scale was pursued.

For electrochemical alkoxylation, the main emphasis was placed on completing the evaluation of the amount of process intensification attainable. A first approach, consisting of the design of a reactor able to work at highly increased operational pressure was evaluated, followed by a second approach, consisting of the design of a reactor allowing for minimisation of the influence of the gas evolved at the counter-electrode on the overall process characteristics by its continuous separation from the reaction mixture. Parallel to this work, a detailed study of the alkoxylation reaction in the ionic liquid solvent was performed.

3.4.1.3 SP3 – Consumer Goods

Plans for the laboratory prototype to deliver results proving that the IMPULSE proposed method is superior compared to the conventional process were pursued. The parameters were optimized to determine a proper foundation for scale-up/scale-out of the process, with respect to the process steps at the demonstration scale, and the feasibility of the process was examined. Two generations of micro falling film reactors (MFFR) were designed and tested, leading to a third generation of MFFR which might have been suitable for demonstration purposes, but which failed due to unresolved issues of fouling. In parallel, a scaled-up version of the SO2-oxydation reactor to be implemented for the demonstration activities was also developed.

For the specific application of perfume encapsulation, empirical quantification of the effects of microreactor operation and design on capsule formation was performed. The mapping of the compatibilities of membranes and perfume components, started in year 3, was pursued. The production of similar capsules in the demonstrator unit and lab reactor at different rates using a scaling factor of five was achieved.

Based on testing with the adapted lab prototypes for the specific application case of controlled emulsification, and based on the ongoing mechanistic studies regarding emulsification processes in general, a pilot scale prototype for the specific application case was developed. Various lab prototypes were designed and tested, and the resulting products were compared. A first version of the reactor to be implemented in the demonstrator unit was developed and built, and further tests of emulsions and vesicle formation were performed at the P&G labs in Brussels.

3.4.2 Cross-sectorial methodological RTD

A final version of the multiscale design methodology, revised in light of experience in operation and integrating outcomes from all of the WPB tasks, was completed and released.

Work was completed on the design and characterisation methodologies involving kinetics, mass and heat transfer, mixing, interconnection and flow distribution.

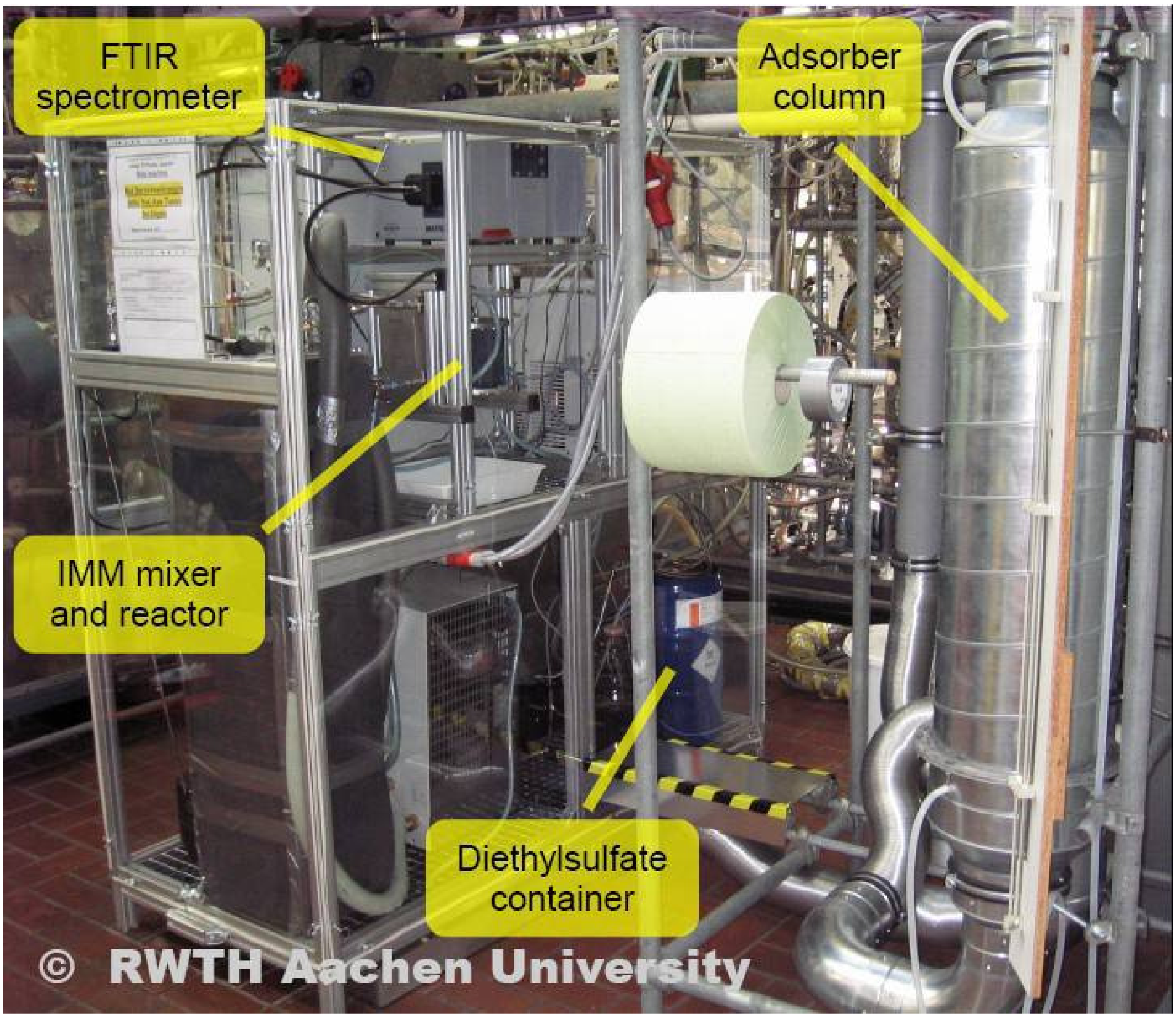
Work continued on the equipment selection methodology, with further development, implementation of the methodology in the equipment database, and population of the database with equipment data supplied by other partners of IMPULSE.

Work was finalised on identifying the industrial needs to meet the challenges of process intensification with multiscale equipment in the area of sustainability, as well as the safety/health, environment and business features required to fill the gaps in current knowledge to respond to those needs.

Work was also pursued on development of tools and methodologies within WPB for the SP tasks, extraction of useful generic information and methodologies from the work carried out in the SP tasks, as well as analysis of the information and transfer to the WPB and WPC tasks for incorporation within the new methodologies.

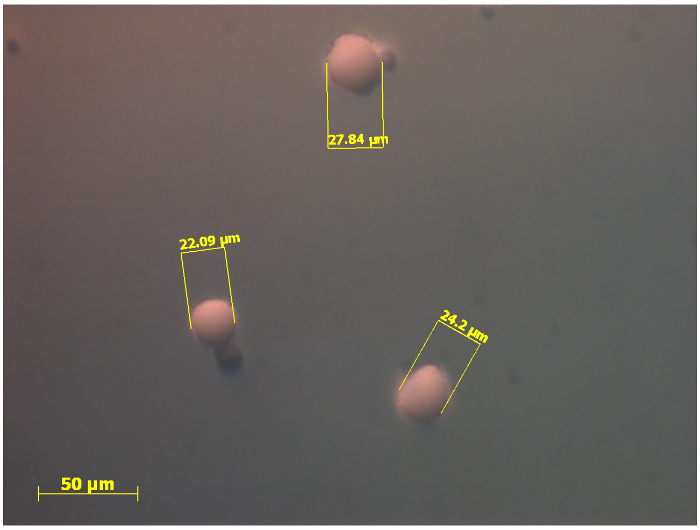
3.4.3 Experimental set-ups

An experimental set-up of the work carried out in year 4 is shown in the pictures below. The picture below shows the Ionic Liquid synthesis demonstrator unit at the RWTH Aachen Pilot Facility



The picture below shows microcapsules produced with the encapsulation pilot setup at P&G.

This encapsulation of a hydrophilic compound cannot be carried out in a batch process and is enabled by the structured continuous approach.



3.4.4 Publishable results, dissemination and the final workshop

Among the publishable results in open fora, the following are year 4 highlights for articles in international journals:

- Di Miceli Raimondi, N., Prat,, L., Gourdon, C., Cognet, P., "Direct numerical simulation of mass transfer in square microchannels for liquid-liquid slug flow," Chemical Engineering Science, 63 (22), 5522-5530 (2008).
- Attour, A., Rode, S., Ziogas, A., Matlosz, M., Lapicque, F., "A thin-gap cell for selective oxidation of 4-methlyanisole to 4-methoxy-benzaldehyde-dimethlyacetal," Journal of Applied Electrochemistry, 38 (3), 339-347 (2008).

The two major conference presentation highlights from year 4 are the following:

- Matlosz M. (2008) "Multiscale design: methods and tools for the sustainable chemical industry of the future", 18th International Congress of Chemical and Process Engineering CHISA, Prague, 24-28 August 2008.
- Löb P., Schütt C., Illg T., Krtschil U., Hofmann C., Metzke D., Kost H.-J., Hessel V. , "Development, realisation and characterisation of a multi-scale reactor set-up targeting 100 kg/d ionic liquid production", 2nd European Process Intensification Conference / Green Process Engineering 2009, Venice, 14-17 June 2009.

In addition, the third IMPULSE Scientific Workshop was held in October 2008 and the third IMPULSE User Group meeting organised in October 2008. The last IMPULSE Newsletters were released in 2008 and early 2009.

4. Promotion and deployment at the end of the project

4.1 The Demonstrators

In total, five IMPULSE demonstration activities were fully deployed by the end of the project, with demonstration units launched in all three of the high–value–added industrial sectors involved : pharmaceutical products, specialty chemicals and consumer goods.

GlaxoSmithKline (GSK), leading the IMPULSE effort on continuous pharmaceutical manufacturing, ran a demo plant on continuous hydrogenation with a fixed bed reactor at its site in Tonbridge, UK. The need of substantial improvements of the overall efficiency of pharmaceutical manufacturing was the main driver for GSK. The expectations on implementing continuous hydrogenations were intrinsically safer processes that provide better selectivity and yields.

Solvent Innovation (SI), an affiliate of Merck KGaA, is a leading industrial partner in the specialty chemicals subproject of IMPULSE and manufacturer of ionic liquids. SI expects higher flexibility in extending manufacturing capacities, higher product quality and safety advantages by going from a batch reactor to a microstructured continuous system with superior heat removal capability. This concept was demonstrated in a 20 kg/day unit at the pilot facilities of partner RWTH Aachen.

Procter & Gamble (P&G), leader of the consumer goods subproject of IMPULSE, launched three demonstration activities: surfactant making, production of perfume microcapsules and production of a liquid fabric enhancer, all performed continuously in microstructured reactors integrated in the P&G pilot flexible microfluidic unit in Brussels. In addition to quality improvements, cost benefits in capital and operating expenditures are expected.

All demonstration activities were accompanied by a number of other IMPULSE partners providing process design and scale-up support (Institute of Microtechnology Mainz, and Karlsruhe Research Center), construction and process control (Siemens) and scientific and operational support by the academic partners (RWTH Aachen, CNRS, ICPF and ICT Prague, and Warsaw University of Technology). The IMPULSE budget reserved for demonstration activities, 1.3 Mio Euro including 0.6 Mio Euro EU funding, was increased by an additional 0.5 Mio Euro contribution of the industrial partners, and the provision of existing infrastructure equivalent to 2.5 Mio Euro.

4.2 The IMPULSE Big Book

An IMPULSE "Big Book" was produced at the end of the project, summarising the essential features of the methodological developments. The Big Book describes tools and methodologies to support multiscale process design for the chemical and pharmaceutical industry. It provides guidance for process designers and offers decision making tools as well as technical protocols (experimentation, equipment design and process control). The aim of the IMPULSE Big Book is to provide a coherent and easily accessible description of the entire methodology to facilitate immediate deployment by experienced process technologists, either chemists or engineers.

Public availability: in the first semester of 2010,

Links: http://impulse.inpl-nancy.fr and www.impulse-project.org

4.3 The IMPULSE Equipment Database

The IMPULSE Equipment Database contains equipment characteristics for various types of structured process equipment (reactors, mixers, heat exchangers, etc.). The equipment database is a guide for process technologists to help select suitable process technologies for reaction, heat and mass transfer duties. The database is provided in Microsoft Access 2003 format and requires the user to have Access 2003 installed on his or her computer in order to use it. A user guide for the database and a set of instructions for equipment characterisation are also provided.

Public availability: June 2009,

Links: http://impulse.inpl-nancy.fr and www.impulse-project.org