Integrated Multiscale Process Units with Locally Structured Elements

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Low–cost engineered components with structural features in the range of microns to millimeters ("microstructured objects") are now readily available in a wide variety of chemically resistant materials such as glass, stainless steel, ceramics, polymers, alloys and graphite (and no longer only silicon). These new microstructured objects offer stimulating perspectives for the development of a new generation of highly original process components and systems for chemical and biochemical application, which can be exploited to improve significantly the quality, safety and effectiveness of industrial production.

Applications of microfabrication technology in microelectronics and micromechanics have already led to revolutionary development in numerous industries, and the structured multiscale chemical devices envisioned here offer the perspective of significant innovation through the use of microfabrication in the chemical process industries as well. Miniaturized components made of robust materials can be exploited to create miniaturised sensors and actuators embedded in process systems and acting on the specific length scales (several tens to hundreds of microns) particularly relevant to chemical processing : boundary layers, transport processes, reaction and mixing zones.

To take full advantage of these advances, however, it is necessary to rethink the way in which chemical processes are designed, built and operated. The concept of structured multiscale process design proposed in the present paper is a direct result of this rethinking.

Microreaction technology : state of the art

Over the last 15 years, substantial research on small–scale structured devices for chemical applications has been undertaken, and a host of academic studies, as well as the seven successful editions of the conference series IMRET (International Conference on Microreaction Technology), have established a solid scientific basis for the fabrication and analysis of individual (generally unconnected) units. A number of reference books are now available [1, 2] as a substantial contribution to the already well established general area of process intensification [3]. Although much of the research efforts in this area have been undertaken in Europe, significant contributions have also come from other regions, in particular the United States and Japan. With the exception of the work of K. Jensen at MIT [4], the American effort on microstructured devices has been mostly oriented toward transportation and military applications, and much less toward chemical production. This is clearly not the case in Japan, however, where an ambitious program on microchemical systems, begun in 2002 and led by J. I. Yoshida, has led to very substantial progress in close collaboration with the Japanese chemical industry [5].

Despite all of these efforts, developments have been dominated by "technology-push", and industrial "market-pull" has not appeared to any extent to be comprehensive or systematic on the part of chemical producers. In a few limited cases, processes employing small–scale structured devices have been utilized for process intensification on the production scale, most notably by Siemens Axiva, Merck, Clariant and Degussa [6, 7, 8]. Apart from Clariant, which processed suspensions, the published examples are quite simple tasks: mixing of miscible liquids, combined with effective heat transport and contact with immobile solids. There has also been some use of other meso–structured devices in production, including intensive rotating contactors [9] and oscillatory baffled reactors [10].

Given the very significant advances in the state-of-the-art in recent years on individual small-scale structured components and devices on the laboratory scale, the major challenge facing the chemical industry is not the further development of individual locally structured units but rather the effective integration of those units into complete production systems. In this connection, it should be noted that the seductively simple ideas of direct "numbering-up" or "scale-out" of microreactor systems initially envisioned in the 1990s are now being brought into question (see, for example, the critical remarks in this regard in [11] and [12].) It now appears clear that the principle of "numbering-up" through direct interconnection of individual small-scale units into large-scale production systems does not really "solve" the scale-up problem as initially intended. On the contrary, "numbering-up" displaces a (well known) chemical engineering problem of process scale-up to a (for the moment essentially unsolved) problem of multiscale process interconnection. In a similar manner, the true impact of individual process-intensification units, such as spinning-disk reactors and similar devices, on whole-process performance (including reactant work-up and related process logistics) has not been completely explored. Basic design principles for process layout and process performance evaluation are clearly needed and these issues will require substantial research efforts in view of the development of a truly generic multiscale design methodology.

As a complement to the detailed research and development efforts, it should be noted that sophisticated methodology for detailed equipment design, interconnection and layout is only of use for industrial application once an initial, preliminary decision has been made to explore new technological options. The availability of approximate, short-cut methods and principles, derived from the more complete, rigorous research results, is an additional challenge for emerging innovation that cannot be ignored.

Examination of the state-of-the-art reveals that for a thorough evaluation of true technological opportunities for the use of small-scale structured components in chemical production, a comprehensive and systematic protocol is required as an aid to decision-making and for ultimate design and exploitation. Whether for retrofit of structured components into existing plant or for new design of future plant facilities, a new methodological approach is an urgent need and a clear contribution to future industrial competitiveness in chemical production technologies. Comparable to a Pinch Analysis for heat integration, or to HAZOP for safety issues, the structured multiscale design methodology should be developed in such as way as to permit reliable qualitative and quantitative techno-economic evaluation of structured multiscale process systems for both existing and potential production processes.

Structured multiscale design: new methodology for process development

Microstructured devices and process components set the stage for a true paradigm shift in the principles of chemical process engineering. Rather than adapting the operating conditions and chemistry to available equipment, the process structure, architecture and equipment can now be adapted to the physico–chemical transformation. Production units can be created by integation and interconnection of diverse, small–scale structured units into large–scale macro–production devices. A key feature of the resulting structured chemical devices is local process control (through integrated sensors and actuators), leading to enhanced global process performance.

Comparison to other methodologies

In order to comprehend fully the interest and promise of the structured multiscale approach proposed here, it is of interest to compare the design methodology of multiscale assembly with other alternative design solutions. In this connection, two contrasting (and in many ways complementary) approaches to industrial process design are worthy of interest :

– traditional scale–up, based on mathematical modeling of large–scale systems

– recently proposed numbering–up, based on replication of small–scale systems

and an analysis of their fundamental basis leads to the new, hybrid approach proposed here :

– multiscale design, characterized by construction of large–scale systems with small–scale inner structuring.

Scale–up by modeling (comparison 1)

The conventional approach to industrialization of chemical processes is generally called “scale–up”. Although in the past, scale–up was frequently performed simply by empirical trial–and–error, the development of chemical and process engineering over the last 40 to 50 years has led to tremendous improvement in design methodology. Among the most important developments has been the growing use of numerical simulation and mathematical modeling of reactor units and production devices in view of their optimization in practice, and in this connection the use of computer–aided design and numerical simulation continues to grow and develop.

For scale–up through mathematical modeling, the large–scale production system is (mathematically) broken down for calculation into a large number of smaller “finite–elements” or “finite–volumes”, but the industrial system itself remains a large–scale device. Optimization is global, based on the mathematical analysis of local physical and chemical phenomena, but without any true attempt to control local operating conditions within the large–scale device.

In the ideal case, scale–up using this traditional approach should include the following steps :

– measurements of reaction kinetics and physico–chemical properties in laboratory devices in order to obtain precise information on rate laws, temperature dependencies, etc.

– calculations of fluid flow, temperature and concentration fields, in standard reactor devices

– choice of optimal conditions taking into account the variations in operating conditions throughout the device, calculated by using the highly precise kinetic information from the laboratory studies, and using sophisticated calculation methods for the large–scale device (such as computational fluid dyamics : CFD).

The clear advantage to this approach is that with detailed kinetic information and accurate modeling of large–scale devices, it is possible to design processes with high performance in standard industrial equipment. It should be noted that if “microreactors” can be used to provide some quantitative information, they can be used in laboratory studies to shorten time and improve knowledge for the scale–up to existing equipment. (It is not necessary to use microreactors for production, however, a significant difference with the numbering–up approach presented below.)

There are nevertheless clear disadvantages for rapid industrial development with the traditional approach. The most obvious disadvantage is that detailed kinetic and physico–chemical property information is required over a large range of operating conditions for accurate modeling, and this information is difficult to obtain rapidly in laboratory devices. In particular, significant quantities of reactants are required and detailed studies to obtain rate expressions can be long and time–consuming.

Furthermore, it is not possible in the traditional large–scale devices of the chemical process industries to control local operating conditions throughout. Hence a compromise must be found between external operating parameters (reactor wall temperature, inlet flowrate, etc.) and process performance. Variations in local operating conditions are not imposed within the device but rather “accepted” from spontaneous distributions within the large–scale system.

Numbering–up by replication (comparison 2)

In the last decade, a new alternative to traditional scale–up has been proposed in the context of “microreaction technology”. The new approach has been coined “scale–out” or “numbering–up” and has attracted considerable academic interest.

With the “numbering–up” approach, the system of interest is studied ONLY on a small scale in so–called “microreactors”, and the final reactor design is simply a multiplication of (interconnected) small–scale devices acting independently. No attempt is made at large–scale optimization. Instead, the optimal functioning point is found for a small–scale device by empirical laboratory studies and then is simply reproduced by “replication” into a larger structure.

The obvious advantages of such an approach are that empirical measurements of “qualitative” performance can be used directly, without recourse to the precise mechanisms necessary for the detailed “modeling” of the traditional “scale–up” approach. The industrial user seeks optimal conditions for running the reactor or chemical device in microreactors, and that information is obtained quickly, with small quantities of reactants in a relatively short time.

It has even been claimed that scale up “disappears” with the “numbering–up” approach, since the macro–device can be obtained (in principle !) by simple multiplication (or replication) of the laboratory microreactors. By interconnecting large numbers of microreactors, macro–production devices should be achievable for which each component micro–unit will run under the same optimum conditions as those found in the laboratory device.

In reality, although this approach may avoid “scale–up” of the reactor itself in the traditional sense, it does not “solve” the problem of scale–up but simply changes the nature of the problem. The true “scale–up” problem results in the numbering–up approach from the clear difficulty of optimal design for the numerous interconnections between reactors, and for the essential connections of the “micro–reactor–assembly” to the “macro–structure” necessary to feed the system.

Upon reflection, one concludes that it is rather unlikely that individual laboratory microreactors will be connected in this way in industrial designs. More likely is that large scale macro–devices will be created with internal microstructuring, and it is not evident that such devices (such as for example multiplate microchannel reactors) will truly operate under identical conditions at all points in the interconnected structure. Numbering–up is therefore not the complete answer to the scale–up problem, but it does provide a stimulating model for a totally new way to design and construct reactor devices.

Among the points that need to be addressed is the fact that even if direct numbering–up could be achieved, it is not clear that it would necessarily be desirable to have the SAME operating conditions at all points in a macro–production device. In fact, when compared to a laboratory device, the “objective” for optimization of a production device is not the same : for a measurement device one is seeking “information”, whereas for a production device, one is not seeking information but rather “performance” for a given throughput of matter and energy. The optimization criteria are generally different.

A laboratory reactor, for example, can frequently be operated at low conversion, whereas an industrial reactor is frequently operating at high conversion. It is therefore more likely that the optimal operating conditions for a true reactor will differ significantly from those found for the laboratory device. Here again, even if numbering–up were possible, it is not clear that one would want to have all points in a reactor operating at the same conditions. It might be preferable to modify operating conditions as a function of position in the macro–structure (see below : local process control).

Structured Multiscale Design : a new hybrid approach

With the multiscale assembly approach, diverse, interacting small–scale units are integrated into a large–scale device that is globally optimized with respect to standard industrial performance criteria. Local sensors and actuators permit local process control of operating conditions (that may vary with position and time) throughout the reactor unit. The extreme diversity of such devices promises particularly rich and innovative designs. The approach is not limited to any particular scale, but with the recent advances in microfabrication on the sub–millimeter scale and the fact that many important transport and transfer phenomena occur on length scales on the order of several tens to hundreds of microns, indicates that the time is ripe to look into new design methodologies that can benefit from the availability of lowcost microstructured components and devices in a variety of materials. These devices could be helpful to realize production on demand (avoid transportation of hazardous chemicals, elimination or reduction of ecological risks) and to speed–up time–to–market for new production processes.

IMPULSE : an industrial–academic research consortium on structured design

In order to advance development of methodology on structured multiscale design, an industrial–academic consortium has been created in the framework of the SUSTECH initiative of CEFIC, the European Chemical Industry Council (see www.cefic-sustech.org). The objective of the consortium research is enhanced performance through targeted, localized intensification.

IMPULSE (an acronym for “Integrated Multiscale Process Units with Locally Structured Elements”) aims at effective, 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 (not simply individual components or unit operations), thereby contributing to significant improvement in supply–chain sustainability. The design concepts and process development tools resulting from the IMPULSE research will be new and unfamiliar. To be accepted in industrial practice, the new approaches must be developed, analyzed, tested and PROVEN on true industrial processes. The collaborative effort of industrial and academic partners is a necessary element for success.

The strategy and rationale of the research is to move from optimization of individual equipment units to whole process design. As shown in the state–of–the–art described above, recent developments of intensified process equipment have mostly been limited to individual unit operations (such as mixers, reactors, separators or exchangers), but appropriate integration of these intensified devices into complete production units has not yet been fully addressed from an industrial perspective.

To respond to this challenge, the IMPULSE consortium proposes to orient research and development toward complete production systems. Design of such systems will involve integration and interconnection of diverse, small–scale structured components into large–scale macro–production devices. The IMPULSE philosophy is to provide intensification locally only in those parts of a process where it is truly needed, and then to adapt interconnection of the locally intensified structures into a global macro–device.

The local, targeted intensification promoted in IMPULSE should enhance process performance by

- facilitating conversion from batch to continuous processing
- increasing safety due to a lower holdup of hazardous components
- providing improved, more reliable and more reproducible quality
- enabling higher yield and selectivity
- reducing resource consumption (energy, reagents, water, solvents)

♦ offering access to new synthesis routes and to on-site on-demand manufacture

while at the same time remaining competitive, cost–effective and reliable. Furthermore, the IMPULSE approach is not only appropriate for new process designs but is also relevant for retrofit of innovative process equipment into existing plant structures and for modular plant designs.

To attain acceptance in industrial practice, it is necessary to provide a number of validated business cases for true commercial processes of industrial interest, in the framework of a multidisciplinary R and D program. Overall results should include :

- proof of principle in several major industrial supply–chain sectors
- validated business models, including technico–economic analysis for each case
- generic design and optimization rules and software tools for their implementation
- decision criteria for appropriate choices of multiscale approaches in practice

The project is structured around three broad subprojects covering products from three major supply–chain sectors : pharmaceuticals, specialty chemicals and consumer goods. Each subproject sector includes one or more market segments. For each market segment, multidisciplinary research teams, led by an industrial producer, identify promising chemical process systems and undertake specific directed research, development and demonstration on those systems with respect to the IMPULSE rationale.

Parallel to this subproject structure, workpackages on generic issues cover the common ground among the subprojects and provide cohesion and integration. Examples of generic issues include instrumentation (sensors and actuators), equipment and control devices, as well as tools, methodology, standardization, design rules and safety.

The following 18 companies and research institutes comprise the current group of partners in the IMPULSE consortium : CNRS (F), Siemens (D), Degussa (D), Procter and Gamble Europe (B), GlaxoSmithKline (UK), Solvent Innovation (D), Britest Limited (UK), Dechema (D), INPL–Nancy (F), RWTH–Aachen (D), UMIST–Manchester (UK), TNO (NL), IMM–Mainz (D), FZK–Karlsruhe (D), INERIS (F), ICPF–Prague (CZ), WUT–Warsaw (PL) and ETSEQ–Tarragona (ES). The IMPULSE Integrated R and D Project, programmed as a 4–year initiative, has now been retained for final contract negotiation for financial support within the European Commission's 6th Framework Programme for Research and Technological Development (see : www.cordis.lu/fp6). The project should begin during the first semester of 2005, following signature of the official consortium contract.

Conclusion

The methodology for structured multiscale chemical process design is an extremely promising area of scientific and technological investigation, rich in theoretical and experimental research and with significant potential for industrial competitiveness. In addition, the new approach should have a lasting impact on the teaching of chemical engineering and industrial chemistry, and should contribute to the attractiveness and acceptability of the chemical process industries as well.

Although relatively easy to imagine in principle, true industrial development and use of the concepts of structured multiscale devices requires further progress in research, combined with corresponding advances in the microfabrication methods required for the construction of the devices themselves. Targeted laboratory and pilot–scale demonstrator units are an urgent necessity in this regard, along with critical technico–economic analysis of potential practical applications. Despite the difficulties, prospects and potential uses for these new chemical devices and production systems are considerable, and concerted research actions between industrial and academic institutions should lead to rapid advances and significant perspectives for their development in the near future. One such action, the IMPULSE consortium, has been founded on this basis in Europe and should encourage development of the necessary methodological tools required for future industrial implementation of the approach.

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