Final publishable summary report (extract from project final report)

Grant Agreement number: 228853-1

Project acronym: COPIRIDE

Project title: Combining Process Intensification-driven Manufacture of Microstructured Reactors and Process Design regarding to Industrial Dimensions and Environment

Funding Scheme: Collaborative project; Large-scale integrating project

Period covered: from 1st September 2009 to 31st August 2013

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1 Final publishable summary report

1.1 Executive Summary

Grant Agreement number: **228853-1**

Project acronym: **COPIRIDE**

Project title: **Combining Process Intensification-driven Manufacture of Microstructured Reactors and Process Design regarding to Industrial Dimensions and Environment**

The ultimate ambition of the EU FP7 project CoPIRIDE "Combining Process Intensification-driven Manufacture of Microstructured Reactors and Process Design Regarding to Industrial Dimensions and Environment" (01.09.2009 - 31.08.2013; 16.9 Mio. € budget) has been to develop **a new modular production and factory concept for the chemical industry using adaptable plants with flexible output for intensified processes under consideration of ecological sustainability and economic viability**.

New modular microstructured reactors have been developed based on mass-manufacturing suited fabrication concepts and brought to production scale **as enabling tool for Process Intensification**.

A further important project element has been to move from conventional to improved processing by exploring deliberately harsh process conditions enabled by the reactor concepts of the project which open up so-called **Novel Process Windows**. The focus was especially on translating low temperature to high temperature processes in order to achieve **increased productivities** due to faster reactions, and to **shift batch synthesis into continuous (flow) synthesis** to gain the advantages of flow processing compared to batch. NPW were successfully exploited in the anionic polymerisation (**high product quality** reached), the epoxidation of soybean oil (**increased space-time yield**) and the supercritical biodiesel production (**opening processing of low-value feedstock i.e. waste oils**).

All main efforts within CoPIRIDE have been grouped around **specific chemical processes as concrete application background**: biomass gasification in view of decentralized ammonia production, biodiesel production, synthesis of a special polymer in a two-step process (the polymerisation itself followed by a hydrogenation reaction), and epoxidation of soybean oil. These have been thereby defined and supervised by industrial partners in the project. Finally in all cases piloting activities have been done for demonstrating central project results also above the level of the improvement of the chemical process itself. Continuous sugar hydrogenation has been followed additionally in a more explorative manner.

Based on but above the level of reactors and processes, **new production plant concepts** have been pushed forward e.g. by Evonik with its **Evotrainer approach as flexible, mobile and low-cost investment plant in container-format** and as tool to **reduce time-to-market** and by the **Flow**

Miniplant concept with a stronger focus on a **modular assembly** and **easy integration in existing facility infrastructures**. Both two new plant concepts have been brought to demonstration stage, the latter one using also the novel developed modular microreactors. Two more, dedicated pilot plants have been set-up and operated: An **oxygasifier for biomass gasification** as preparing step for decentralized production of ammonia or other chemical products and a **pilot-plant for harsh process conditions in view of biodiesel production from waste-oils under supercritical process conditions**.

Quite unique to the CoPIRIDE project has been that from the beginning simplified **cost analysis** and **Life Cycle Assessments** have been performed to judge different processing/production options as guiding and early-bird decision tool for project works to focus on **ecological sustainable and economic viable processes**.

Project contents and results have been **disseminated** by the project partners by **more than 250 activities** including **publications in peer-reviewed journal, oral and poster contributions to conferences, newsletters, participation in joined activities of other EU projects in the field and the organization of a public dissemination event.** With regard to **training**, joint training materials have been elaborated and a summer school has been organized.

Project works and results lead to **6 patent applications**. Two of them are not published so far and so details have to be kept confidential; these two patent applications relate to **novel process options** for the polymerization case study. Two filed patents concern the protecting of **CoPIRIDE microreactor concepts and their fabrication**. Finally, two patent applications around **ammonia production by integrated intensified processes** starting from syngas generated by biomass gasification and using a **novel catalyst system** and special membrane reactors for syngas cleaning have been filed. **Besides the general advancement of knowledge** generated in general especially by universities and research organizations, **7 exploitable results** have been identified by the project partners.

The frame of the EU project allowed bringing **multi-disciplinary** expertise in **universities, research centers and enterprises** from different EU member states (in total 16 partners) together in a **complimentary** manner to tackle the addressed challenges successfully and fostering **EU wide collaboration**. Both scientific and industrial partner benefited through the **uptake of scientific achievements into business** and the increase in interdisciplinary interaction to foster the generation of new knowledge. The project is so also contributing to the **transition to a knowledge-intensive European industry**. Especially the new plants concepts contribute to the move towards **adaptive production** in Europe. The results of the project will foster especially the **competitiveness of European chemical industry including SME** and contributes to **more sustainable European chemical production**.

Project web site: http://www.copiride.eu/

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1.2 Summary description of project context and objectives

1.2.1 Problem situation

Starting point of the initial project considerations has been on one hand that the **chemical market** is **under considerable cost and ecological pressure**, especially in view of the new emerging markets and production capabilities in Asia and on the other hand that it is a promising approach to **improve competitiveness by** the concept of **process intensification**.

Process intensification (PI) means the development of entirely new concepts for process steps and equipment in contrast to process development, relying on performance improvement of existing concepts. Process intensification demands for strong interdisciplinarity (chemistry and catalysis, applied physics, mechanical engineering, materials science, electronics, etc.). The **state-of-the-art of PI in chemical industries**, especially when dealing with micro process technologies, was **at project start** considered as follows:

- Several demonstration runs in **dedicated, home-made pilot plants built in industrial laboratories using stock lab equipment**. Examples to bridge to production in **heavy pilots**, typically constructed of fabricated metal on dedicated concrete slabs and costing millions of dollars, are **rare**.
- **Microstructured reactors** are available **for the laboratory based pilot plants**. **Production-targeting microreactor** designs with extended outer dimension, high degree of numbering-up and system integration are **rare in fine chemistry**.
- **PI** is **demonstrated for the stage of reaction**. **Missing is a holistic view, taking into account the whole process development cycle**, including separation, process control, safety installation, and plant approval up to production. This requires identification of additional PI drivers for the whole development chains.

1.2.2 Main objectives and envisioned breakthroughs

CoPIRIDE has followed the definition of process intensification as given above and **has targeted** to provide **entirely new concepts – both for process steps and equipment/plants**. CoPIRIDE has strived for **improving technology** at the **catalyst – (reactor) fabrication – reactor – plant – process** level **up to a production stage**.

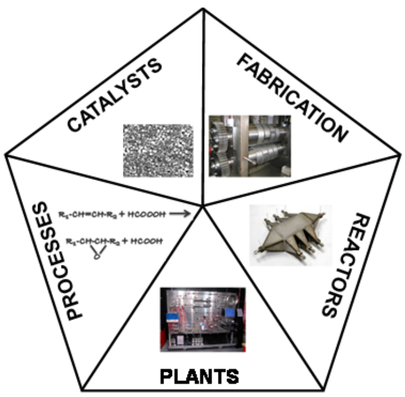


Fig. 1: Decisive technology fields in CoPIRIDE to reach process intensification and whole development.

These five research areas have been seen as keys to **achieve PI** and to provide a strongly **interdisciplinary and holistic development**; thus covering not only the initial steps of process development, but including a view on the **whole development** with process control, plant approval, and operational duties. CoPIRIDE specifically envisioned the following breakthroughs:

- New, IP protected modular production & factory concepts
- Adaptable plants with flexible output
- Production-scale & mass-manufactured microstructured reactors
- Integrated reactors and processes
- Reduction of resources and costs & improvement of eco-efficiency

CoPIRIDE also has targeted the **broader uptake of PI know-how and technology** in view of a broader European geographical spread, an implementation in SME aside larger companies, and transfer from chemical sector to other branches (food and biofuels).

1.2.3 Concept

The project has focused on the following working areas to achieve its objectives:

- **Introduction of new-generation catalysts** with high catalyst activity **to microchannels** to support novel process windows tailored for micro process technology based process intensification.
- **Expansion**, standardisation, and modularisation **of microstructured and PI reactor toolbox**:

  Milli and microstructured reactors are due to their unique properties especially with regard to mass and heat transfer a preferred type of apparatuses for PI. In a broader sense this includes monoliths, foams, mini fixed bed and capillary/tube reactors. In view of industrial processes further development has been seen necessary especially picking-up the following aspects:

  - The quest for Novel Process Windows, particularly for high-p, T applications, demands for new joining techniques for microstructured components, such as brazing or soldering.
  - Design flexibility to address different requested functionalities.
  - Modularity in design on several hierarchies of microstructure integration defined by the microfabrication approach (e.g. multiple channels on a plate, stack of plates, combination of plate stacks)

- **New, mass-suited microfabrication techniques**:

  The quest has been here mainly in developing cost-efficient mass manufacturing suited techniques and also in approaching manufacturing techniques for larger plate dimensions (for reactors for higher throughput and longer residence times).

- **New plant concepts**:

  Due to the specific challenges of the application considered in the project, a two-fold approach has been taken in view of plant concepts (see Fig. 2)

  Modular mini-production plant platforms: Pilot plant is a relative term in the sense that plants are typically smaller than production scale plants, but are built in a range of sizes. Some pilot plants are built in laboratories using stock lab equipment. Others are constructed of fabricated metal on dedicated concrete slabs and cost millions of euros. The project has aimed at the latter concept, but intended to provide new, modular concepts. In the following the term mini-production plant is used for this in project context to distinguish them from simple laboratory-based versions and to outline that finally these can directly be used for chemical production.

Complete modularity of an entire plant is an ideal, often beyond real-life industrial interests. In project context, plant modularity part by part has been envisioned addressing different **degrees of modularity** concerning a) process development from **lab to production** for one reaction, b) a **choice of reactions**, and c) the **whole process** including separation, control, and safety.
Accordingly, the **idea** has been **to target for a joint plant platform** which serves as multi-purpose infrastructure to integrate individual reaction and separation modules. Generally, the concept has been to have **functional modules in block format**, one each for feed (pumps), reactors, peripherals / balance-of-plant components, process control, safety installations, and on-line analytics. In another approach, all the latter with the exception of feed and reaction are encompassed in a common frame with **cells as substructure**, e.g. using a container-like format. A technical and IRP base for this was laid already before the project by the Evotrainer platform approach of Evonik Industries AG which has nevertheless asked for further development and concretisation. This platform comprises standardised basic logistics such as for the feed system, process control, on-line analytics, and data acquisition. It has been aimed in the project to decide on the specific application requirements between the two approaches (block-based or cell-based) or to even integrate both approaches into a united format.

Dedicated plants: Besides having unified mini-production plant (for the fine chemical application examples), the testing of new advanced process concepts such as supercritical processing or advanced process integration demands for special reactor and plant development. For these cases, modularisation has not been seen as appropriate approach in the project. So, dedicated pilot plants have been followed here.

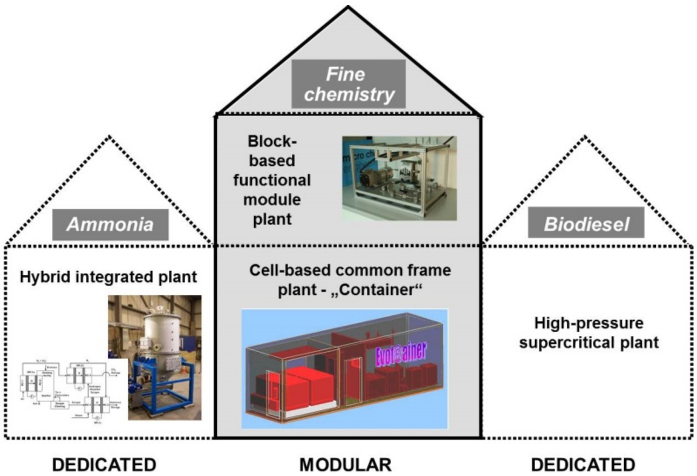


Fig. 2: *Plant concepts within the CoPIRIDE project.*

- Dimorphic process & plant approach using tailored processing ("**Novel Process Windows**"):

There is always interdependency between process and plant design which is called dimorphic here. An improved process will require new tailored plants; availability of higher-performance reactors and plants will enable new processes. But often with the current processes, the potential of microreaction technology is by far not fully exploited. Besides improving processes, one can even reach operation conditions which are not accessible or which are at least unscaleable with conventional equipment. The idea to deliberately explore harsh or otherwise very unusual process conditions for process intensification of chemical reactions is termed "Novel Process Windows" concept. The general approach is to operate at conditions which considerably speed up conversion rates, while maintaining selectivity. This is achieved, e.g., by step-change increases in temperature, pressure, or concentration, or by simplifying the process flow and protocol via fast, simultaneous carrying out of process steps as well as function integration. It has been the ambition of the CoPIRIDE project to put the exploration of "Novel Process Windows" in the focus of all process development activities.

- **Holistic LCA and cost calculation** for the entire development chain – Ex-ante and after demonstration:

  The project has targeted for economic viable and sustainable process/production solutions. **Ex-ante** cost analysis and LCA have been planned **from the beginning** of development works to judge different process options and to guide development direction. Finally, **complete** cost calculation and LCA have been planned **at the end based on the results** of the piloting activities.

- **Industrial pilot activities** demonstrated by medium and small companies.
- **Dissemination, exploitation, training** and **education activities**.

As additional structuring element in the **project works**, all main efforts within CoPIRIDE have been **aligned around specific chemical processes** with their individual challenges. Furthermore for all the main chemical processes defined and supervised by industrial partners, piloting activities have been envisioned to demonstrate central project results also above the level of the improvement of the chemical process itself:

- **Biomass gasification in view of decentralized ammonia production** (industry partner: ITI Energy Ltd.);
- **Biodiesel production** (Chemtex Italia srl);
- **Synthesis of a special polymer** in a two-step process (Evonik Industries AG);
- **Epoxidation of soybean oil** (Mythen S.p.A.).

Sugar hydrogenation with a more explorative view into direction of food industry and without a specific industrial partner behind has formed a further chemical process considered in the project.

The project concept is illustrated and summarized in a condensed form in the following figure.

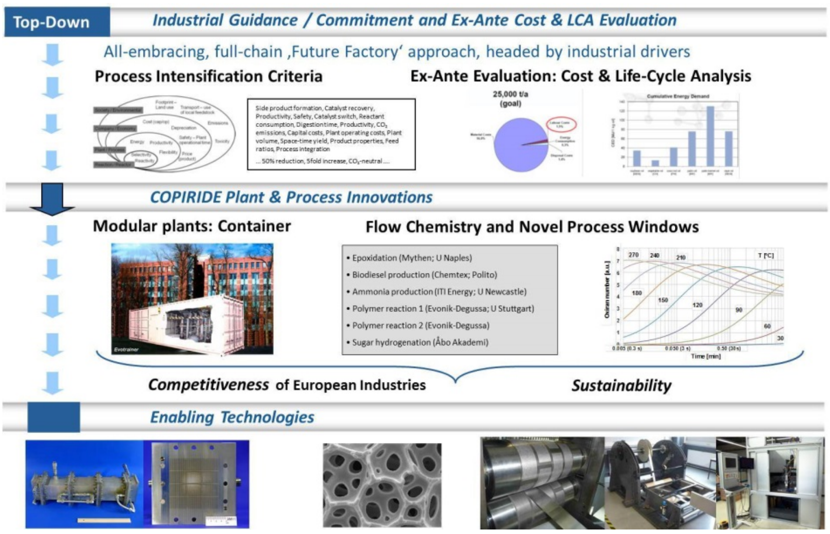


Fig. 3: *Representation of major traits of the CoPIRIDE project concept.*

1.3 Description of the main S&T results

The main science and technological results have been grouped into four case studies around chemical processes up to corresponding plant concepts and three focus topics (microreactor concepts and fabrication; Novel Process Windows; cost analysis and LCA) in the following seven chapters to allow an easy and illustrative insight into the project works and results. The chapters have been provided in most cases by the lead partners. Some refining has been done by the coordinator.

1.3.1 Case story: Evotrainer based chemical plants – accelerating innovation

Introduction - Chemistry in a container based universal infrastructure allows small-scale production under real-life conditions

The advantages of compact small-scale systems are obvious: compared with a commercial-scale plant, the investment costs are relatively low and the market risk is significantly smaller. The experts can develop a process independently of the site where subsequent production takes place, which saves valuable time. Container chemistry produces substances to the exact requirements of the market and customer. Moreover, it is no pilot plant in the traditional sense, because it later serves as a "real" production plant, often without extensive modification. Small-scale enables fast and simple capacity adjustment: if demand rises more than expected, production is expanded to several containers or can even be directly transferred to a large-scale plant. This approach splits both investment costs and risks - not an insignificant factor for a company.

Above all, however, small-scale plants shorten the time from idea to market entry. Laboratory development and basic engineering -planning phases that are otherwise strictly separated - can take place simultaneously. This is because the container is not only the place where the new process is developed but where production is also planned. Make something small from something large - it sounds simple, but it poses a real challenge for planners and developers. A reaction in the glass flask may work perfectly, but will it work just as well in continuous production with pencil-thin reaction tubes?

Even the engineering is anything but trivial: space is limited and, therefore, valuable. Engineers must accommodate all the functionalities of a chemical plant in a space not much larger than a garage. Small is beautiful - but only if some central challenges can be overcome. On one side, small-scale production has specific technical requirements. Small volumes often mean acceleration of the mass transfer. Thus, the measuring and control technology must be far more sensitive than in large-scale plants. Acceleration of processes also places higher demands on the measuring technology. On the other hand, the short routes in the container enable far better heat integration.

Small scale means a paradigm shift

Small-scale bridges the gap between laboratory, pilot plant, and real production. A container-based process is usually designed to be a continuous process, not least due to the less volume of the required devices. For chemists, this is a true paradigm shift, since a laboratory or pilot scale production process is usually developed as discontinuously operated process. A complete chemical plant in an extremely small space! How does that work in reality? Finding an answer to this question has been one of the objectives Evonik experts, in cooperation with other companies and several universities have been pursuing since 2009 in their work on the EU research project CoPIRIDE. As part of the project, Evonik has partnered with the universities of Stuttgart (Germany) and Eindhoven (the

Netherlands) and the Institut für Mikrotechnik Mainz GmbH (IMM) in Mainz (Germany) on the development of a third-generation container. The unique feature of the container will be its ability to be used anywhere – see Fig. 1. The supply system for water, process gases, electricity, heat, and data lines is designed in such a way that, theoretically, any chemical reaction can be run in it.

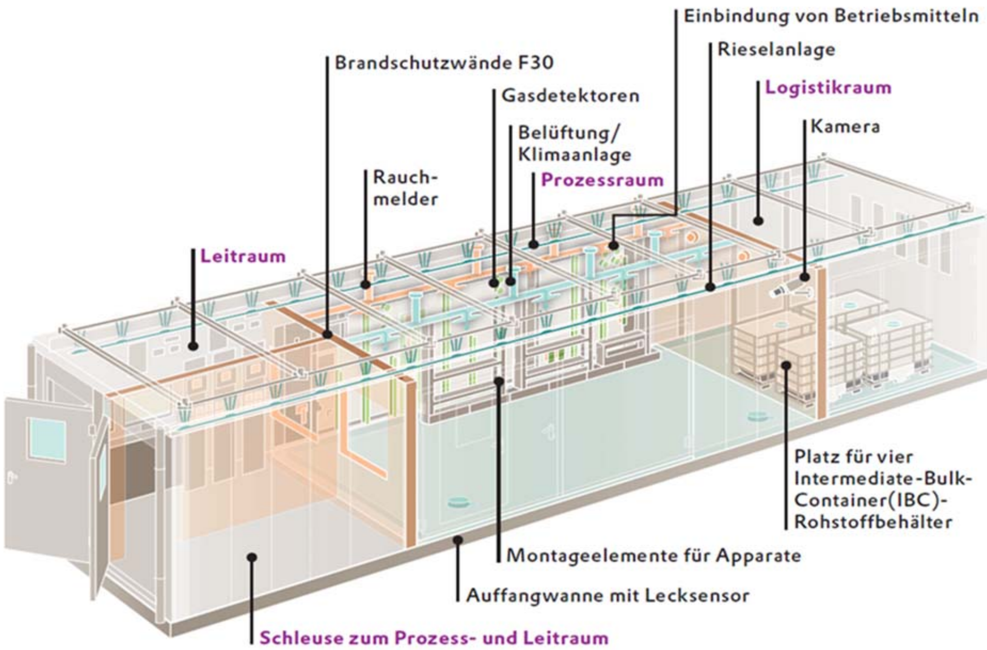


Fig. 4: *The Evotrainer mini-factory. Measuring only 3 by 12 meters, the container holds everything needed for production-reactors, process control technology, IT modules, storage space for feedstocks, elements for constructive fire protection, escape doors, and catch basins (Source: Evonik).*

A universal infrastructure standard is the key to success

The beauty of the idea lies in its versatility. For example, a complete chemical plant, but also only one single reactor for a special downstream processing step can be integrated. High-pressure technology, comprehensive safety technology, and an ultra-compact design are particularly important for the processes in the CoPIRIDE Project. The project also does demonstrate that the approach can be used to run reactions safely and easily under highly critical process conditions.

Regardless of the process, modularization and standardization plays a key role in small-scale plants. A module comprises a particular plant area or component with certain technical requirements. As a rule, modules are standardized and prefabricated structural elements or component groups that accelerate the planning and construction of a plant and reduce the costs of operation. Small-scale design and modularization are mutually beneficial. A module may always be less than optimal because of the compromises one frequently makes between requirements, but it can be available fast and at a reasonable price. If the container process is modular, the processes can be modified or swapped quickly. Inversely, modularization is advanced because function and design repeat themselves in small scale plants.

EvoTrainer based chemical plant

How the time from product idea to final production process can significantly be reduced is shown in the following. The core of the idea is to centralize the development process in one infrastructure. With this, knowledge about the process as well as all know how around the process generated (e.g.

safety issues) were cumulated at one place. These circumstances make it possible to avoid interface problems. Fig. 5 shows in sketches the main steps of bringing a new product idea to market:

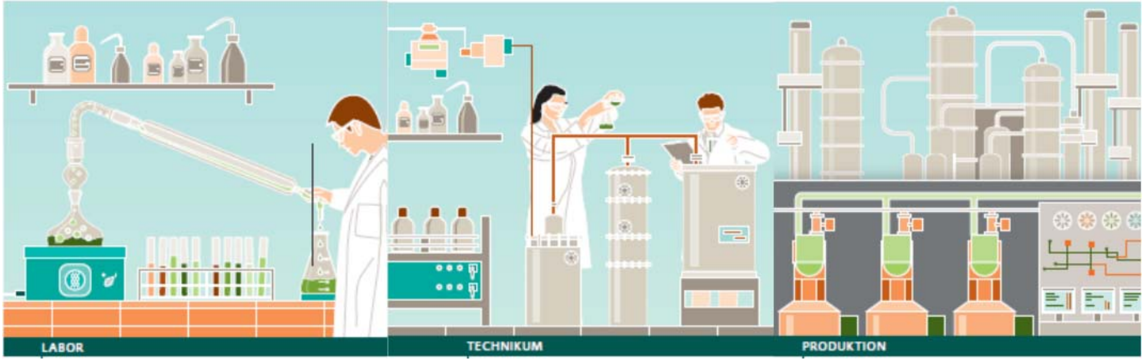


Fig. 5: *The main steps of bringing a new product idea to market: from lab to production (Source: Evonik).*

Now, all these steps can be carried out in the EvoTrainer environment. The EvoTrainer is the mobile small-scale production facilities from Evonik Industries AG. Behind the catchy name is a concept that could thoroughly transform the (specialty) chemicals area.

Fig. 6: *The mobile EvoTrainer 3G: Infrastructure for product development from lab to production (Source: Evonik).*

Within CoPIRIDE two EvoTrainers were transported to Evonik Hanau side, where the two step synthesis parts for the production of a specialty polymer were integrated. After this, both were transported to Evonik site in Marl (Germany). The brand new third generation EvoTrainer plants were put into operation in summer 2013 – which is one of the main outcomes of the CoPIRIDE project.

In line with the proposed approach for new products, Evonik Industries considered before building a large-scale facility, to test the process under real-life conditions in an EvoTrainer environment and develop it to full maturity there (see Fig. 7). One elegant advantage of the EvoTrainer concept is to work with the same kind of equipment and processes as those found in industrial scale facilities. Even the process control system is the same. This makes the potential jump to a large-scale facility later on so much easier, faster, and more secure. The machine operators are trained directly on the real-life systems so that they can learn as effectively as possible.

The EvoTrainer significantly cuts planning costs because all required infrastructure has been thoroughly planned and is ready for installation. The process engineers proudly call it the "150-percent infrastructure." The facility is divided into four areas: the process room, where production takes place; the airlock through which the process room is entered when the system is in operation; the control room, which houses the process control technology; and the logistics room for objects such as raw material containers. Basic equipment includes a process control system that is used to the system's operation from the control room. The EvoTrainer can also be connected to an existing control station. Another standard feature is a collection pan with integrated sensors that trigger an alarm if there is a leak. The ventilation and air conditioning systems are installed in the ceiling. The exhaust air is regulated by the external exhaust air management system. Partition walls can be set up in the EvoTrainer to create EX zones that protect against explosion risks. The outer envelope has an F30 fire-resistance rating and hermetically seals the container. Depending on needs, the system can be fitted with sensors that detect specific air-borne substances or with cameras that scan the interior so that workers do not have to enter. A sprinkler system can be installed to extinguish fires or deal with hazardous gases. The EvoTrainer is supplied with electricity, air, process steam, raw materials and auxiliary materials from outside. Exhaust air is channeled outside the facility. The interfaces for the supply systems are hidden behind a rollup shutter in one of the long sides of the container.

Fig. 7: *EvoTrainer plant at Marl side. (Source Evonik)*

"Rent a plant" as business model

The specialists at Evonik are also developing an interesting new business model for the Group's business units: the rentable production plant. The idea for Rent-a-Plant® is that the teams of experts at the Process Technology & Engineering Service Unit, together with the respective R&D departments from a business line, develop and construct the process for small volumes. After start-up testing, the finished production plant can then be transported to the desired production site in the Group - wherever that happens to be. If the container is no longer needed - for example, because a large-scale plant is required - it can be returned and equipped for the next process. The demand for a flexible technology for producing chemical products is growing. Small-scale plants allow a company to market a new product earlier. Product and process development are accelerated, and the financial risk is minimized. Flexible and mobile compact plants adjust to demand and the customer.

Summary

This case study shows (exemplary) the broad application potential of the EvoTrainer approach for process industry sectors e.g. in chemical intermediates, specialty polymers and other consumer products. The project results can be generalized on the whole field of anionic polymerization (e.g. rubber) and show the outstanding production concept based on micro technology as well as the

EvoTrainer plant technology. The capacity can grow with the market, and the plant allows up-scaling without retrofitting. They can also be located almost anywhere. Mobile compact small-scale plants also enable production directly on the customer's site - the Evotrainer can produce wherever the economic conditions are most favorable. The concept allows accelerated innovation cycles. This is a key advantage particularly for fast growing "green" technologies, since the implementation of technical advances is far faster than with classical large-scale chemistry. The chemical industry in Europe has lived off mass-produced chemicals for a long time. But times are changing. Pharmaceutical companies are not the only ones obliged to supply more new, innovative substances earlier than ever before - substances that may be needed in relatively small quantities but that have interesting properties and add a great deal of value. Miniaturized chemical production opens up paths to flexible, efficient and resource-friendly production that meets the growing demands of a globalized marketplace, accelerates innovation, and also gives large corporations a highly promising way to react flexibly to changing conditions.

1.3.2 Case story: Multi-purpose flow chemistry platform for PI by micro process engineering

Close proximity to markets, attractive procurement (raw materials, energy), an enabling administrative environment (taxes, subsidies, conditions, approval procedures) etc. traditionally form the basis for a promising chemical industry. For highly advanced sites factors like intensive cooperation between science, research and industry, high labour force, excellent infrastructure, networking and sustainable production possibilities increasingly get important. According to recent studies the future competitiveness of enterprises will show-up exactly in this areas: As most promising strategies the development of innovation, the focus on specialty chemicals, a large variety of the production portfolio to balance fluctuations in demand, increased efficiency and optimized raw materials (renewable raw materials where it is technically, economically, environmentally feasible and meaningful in social terms) is seen in many market forecasts like Roland Berger, prognostic, accenture, Chamelot etc.

With regard to system engineering the CoPIRIDE objectives exactly fit this need to improve the efficiency and sustainability and to strengthen innovation and competitiveness in Europe were pursued and achieved. Therefore, Microinnova Engineering GmbH had taken up the approach to develop a multifunctional and multi-purpose flow chemistry platform with the integration of process intensification by micro process engineering.

Following this vision Microinnova Engineering in close collaboration with the project partners developed a concept of a modular system enabling future-proof production particularly for specialty and fine chemicals: Flexible, sustainable and highly productive. The integration of intensified processes and high-performance reactors which were also developed by the partners during the project were in focus, in addition special emphasis was put on a most universal application and standardization of the production system.

According to the special requirements like high flexibility and short development times in the field of fine and specialty chemicals a special system was developed that is suitable for process development at the laboratory at a small scale and for production of bigger quantities at a bigger scale. This means that from the very beginning series of tests and experiments in the laboratory are executed under almost the same process conditions as later in production and generated data are available for process simulations and valid for direct up-scaling.

The principles to reduce complexity of systems by modularization apply to various disciplines of natural sciences, mathematics and engineering as well as to sociology and economics. So the innovative manufacturing system was based on modularity and the consideration to summarize functionally directly related process steps and necessary apparatus, sensors and other components to form a structural unit, which is then available as a standardized element for setting up an entire plant. When selecting the components for each module it was checked that they are available for larger throughputs in a suitable manner to ensure the direct scalability.

The so-designed system is called Flow Miniplant. It is a set-up of 4 modules and an integrated control system (Fig. 8). The Flow Miniplant allows a flexible adaptation to new or changed processes in two ways: In case of differing number, sequence or type of process steps a simple adding, removing, or replacing of relevant modules can be made (inter-modular flexibility). In case of new or modified process steps, individual components of a module can be replaced (intra-modular flexibility). For necessary adaptations in case of big changes of temperature and pressure ranges, viscosities, pH values, etc. complete modules for exchanges are designed. The respective connections for the transport of materials are standardized and can be easily connected with hoses or pipes.

In the development of the modules it was also an aim to keep the dimensions small enough, so that a Flow Miniplant for process development as well as one for the production of small quantities can be operated in commercial fume hood. Thus, the use of existing infrastructure to meet required safety regulations is possible.

Within CoPIRIDE the Flow Miniplant concept was applied for the epoxidation of soybean oil (leading to epoxidised soybean oil, ESBO). ESBO is one of the most common oleo chemicals primarily used as a plasticizer replacing Di-Octyl Phthalate (DOP) in plastic materials (in closure gaskets used to seal glass jars, as a stabilizer to minimize the ultraviolet degradation of PVC resins in baby food jars, fillers, paint and lacquers, adhesives, printing inks and packaging etc.) For CoPIRIDE a new process for the epoxidation of soy-bean-oil has been developed and for testing the first, fast and highly exothermic reaction step a modular Flow Miniplant was built (Flow sheet see Fig. 9). It was shown successfully that the critical first part of the reaction can be effectively controlled.

The core of the system is the reaction module equipped with the high end microstructured reactor developed by IMM and the corresponding sensors, control systems and additional heat exchanger. In accordance with the process requirements two new feed modules have been developed for pumping and conditioning the reactants or catalysts, solvents, cleaning substances respectively at a defined mass flow pulsation free into the reactor module. For ESBO it is soybean oil and hydrogen peroxide. One of the feed modules is equipped with a micro angular gear pump and the other with a plunger pump. In front of the pump is a three way valve that enables switching from reactant to flushing medium. Filters protect the pump from solvents. After the pump there is a safety valve to protect the plant from pressure overload. A pressure measurement and the mass flow measurement are used for controlling the speed of the pump. A check valve prevents backflow. One ball valve is used to shut off the module, one is used for purging. In the following, the characteristics of one of the feed modules are given.

Flow range (gear pump):	3 – 20 l/h
Permissible pressure:	0 – 20 barg
Permissible temperature:	10 – 40 °C
Material:	Stainless steel 316, 316L
Dimensions:	450 mm x 600 mm x 425 mm (W x D x H)
Weight:	30 kg

The last module of the system is the product module. It is used to keep a defined pressure upstream the module and for cooling or warming the finished product to room temperature. Behind the heat exchanger there are temperature measurement, pressure measurement and a back pressure control valves. The first three way valve can be used for taking samples and the second one enables switching from the product vessel to the waste vessel. In the following, the characteristics of the feed product module are given.

Flow range:	1 – 10 l/h
Permissible pressure:	0 – 20 bar
Permissible temperature:	10 – 50 °C downstream heat exchange
	-30 – +200 °C heat exchanger and upstream
Material:	Stainless steel 316, 316L

Dimensions:	450 mm x 600 mm x 300 mm (W x D x H)
Weight:	25 kg

Five more modules have been fully engineered (feed modules for formic acid and sulphuric acid and a reaction module for generating performic acid, for epoxidation and for providing residence time) but were not realized until now due to a drop out of one project partner. For piloting of the ESBO process at IMM (see Figure 10), IMM assembled a suited feed module for the acid mixture and a reaction module fitting together with the realised modules of Microinnova.

The main functions of the control software are the plant control, visualization, alerting, logging of the measured values and reporting. The software has been developed in such a way that it is easy to use and has a high flexibility. Every module is displayed separately and has its own software package.

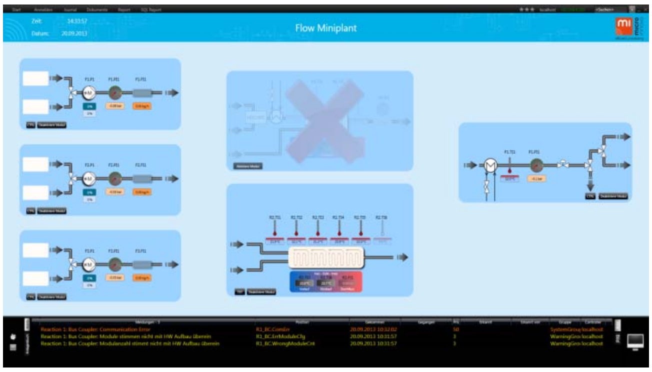


Fig. 8: *User Interface of the Flow Miniplant.*

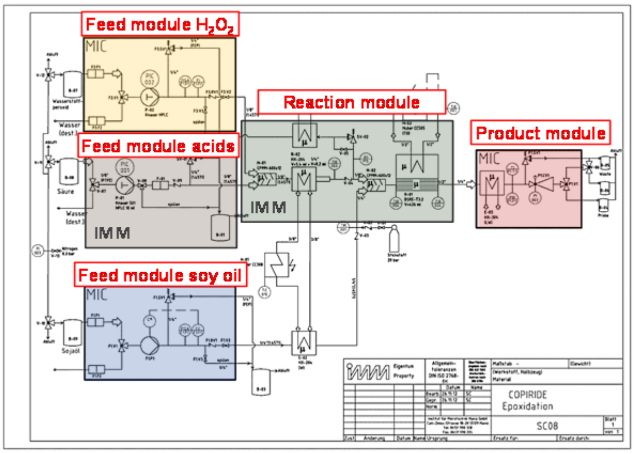


Fig. 9: *Flow sheet of the Flow Miniplant as used for epoxidation pilot trails.*

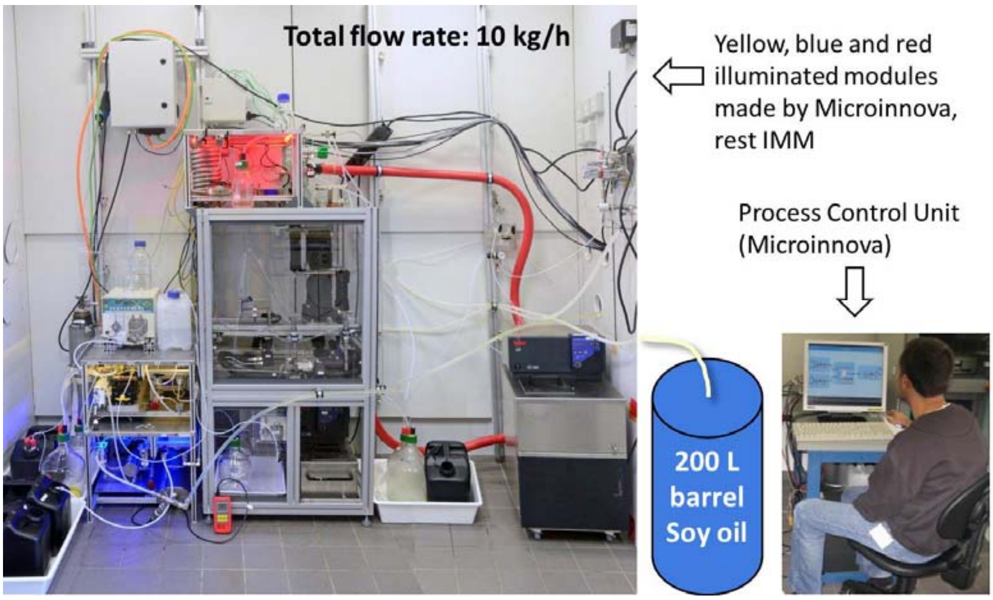


Fig. 10: *Piloting set-up for the epoxidation of soybean oil at IMM.*

To demonstrate the multi-functionality of the Flow Miniplant it has been modified for a propoxylation after piloting the epoxidation as additional demonstration activity with an external partner. Microinnova carried out the adaptions of the plant and of the software settings, the maintenance and has supported the trials. The system allows for a safe, fast and precise processing of fast liquid-liquid reactions.

The switch from conventional to continuous processing (see Fig. 14) and micro reactor technology resulted in many benefits:

- reduction of safety risk
- elimination of critical deviations of batch qualities
- elimination of exhaust gas

Educt and catalyst have been premixed in a static-mixer and heated. This fluid is mixed with the propylenoxid in a special micromixer and reacts in the IMM microreactor (Fig. 12). The heat of the exothermic reaction has been lead away by a cooling thermostat. With the pressure control valve the required pressure could be adjusted.

After the successful completion of the tests at a chemical company the first commercial Flow Miniplant has been built (Fig. 11 – Fig. 13) and sold directly to this strongly impressed manufacturer (plant at customer’s site see Fig. 15).

Fig. 11: *Stack of feed modules (Source: Microinnova).*

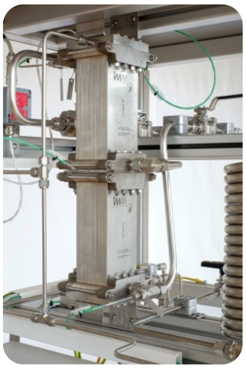


Fig. 12: *Reaction module with high-end modular microstructured reactor by IMM (Source: Microinnova).*

Fig. 13: *Commercial Flow Miniplant for fast l/l reactions like epoxidation and propoxylation (Source: MIC).*

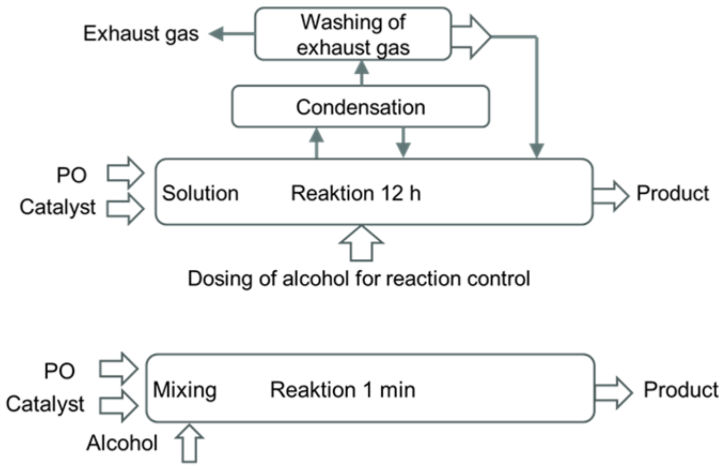


Fig. 14: *Conventional propoxylation (upper scheme) vs. intensified continuous propoxylation (lower scheme).*

Fig. 15: *Trials and commercial continuous propoxylation (Source: Microinnova).*

1.3.3 Case story: Biodiesel from waste oils under supercritical process conditions

Process intensification quite often entails extreme operating conditions, indeed far away from conventional ones (Novel Process Windows).

The reason for this process intensification was, in the biodiesel case-study faced in COPIRIDE, the profitability increase of biodiesel production, through the use of cheap feedstock, via super-critical conditions.

Hence, biodiesel production cost is strictly related to feedstock price, especially vegetable oil, which represent over more than 70% of total production cost. The constant growth of biodiesel production results in a strong demand increase of vegetable oil, which consequently leads to a sharp increase of this material price. At the current vegetable oil price, the production cost of biodiesel is quite higher compared to common petroleum based diesel. Furthermore vegetable oil is used for human feed, so its use raises ethic issue and reduce social acceptance. These reasons drive to the need to find different feedstock, less expensive and more eco-friendly. The use of waste vegetable oil can be the solution, because its use gives a double benefit, as it provides a cheap feedstock, and also allows a waste, the used vegetable oil, to be valorised reused. Supercritical conditions can lead to satisfactory biodiesel yields even in presence of waste oils.

Currently, biodiesel is produced by oil trans-esterification with alcohols in large batch reactors; in this project, the biodiesel synthesis was driven either at super- or sub-critical conditions.

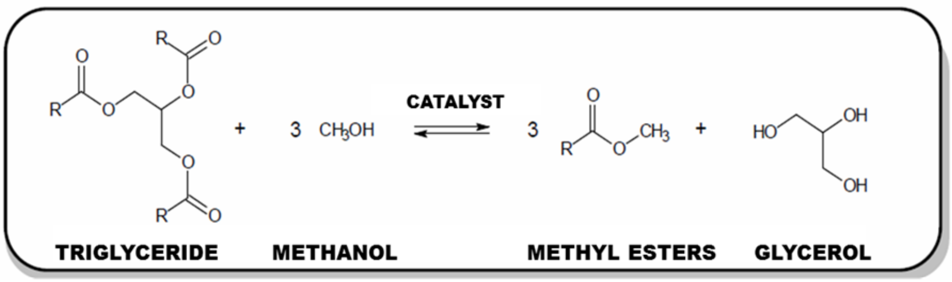


Fig. 16: *Biodiesel (Methyl Esters) synthesis reaction.*

The key point of this technology is the possibility to avoid the use of homogenous base catalysts (KOH), thus allowing to employ waste oils as feedstock (which is currently the most profitable application for biodiesel), having a high content of free fatty acids. Similarly, supercritical conditions avoid the use of liquid phase acid catalysts (H_2SO_4), thus reducing its recovery in the separation section, and the associated energy cost. For what concerns the heterogeneous processes for biodiesel production, it has to be pointed point out that the unique commercialized solid catalyst process is the one developed by the Institute Français du Pétrole, which was designed for the trans-esterification of refined oil, with very low free fatty acid content.

Within the COPIRIDE project, Politecnico di Torino and Chemtex Italia have initially gained experience of the complex phenomenology occurring in the supercritical trans-esterification with methanol: the development of reactor line, based on a stirred tank autoclave, proved that quite high conversions can be achieved via this route at residence times lower than 10 minutes. A further lowering of the residence time was enabled by the use of a catalyst (MgO) with limited leaching. This catalyst was found active both under super- and sub-critical conditions.

The second phase of the activity was devoted to a proper development of an intensified process in terms of performance and control at a pilot-scale, with a size being more representative of the issues occurring in real plants rather than at a laboratory scale. To this end, a mini-plant having a productivity of about 0.1 liter of FAME per minute was conceived, designed and erected (Figure 17). The mini-plant hosts not only the reactor, but also the heat recovery means, since heat integration management is crucial for the efficiency of the plant.

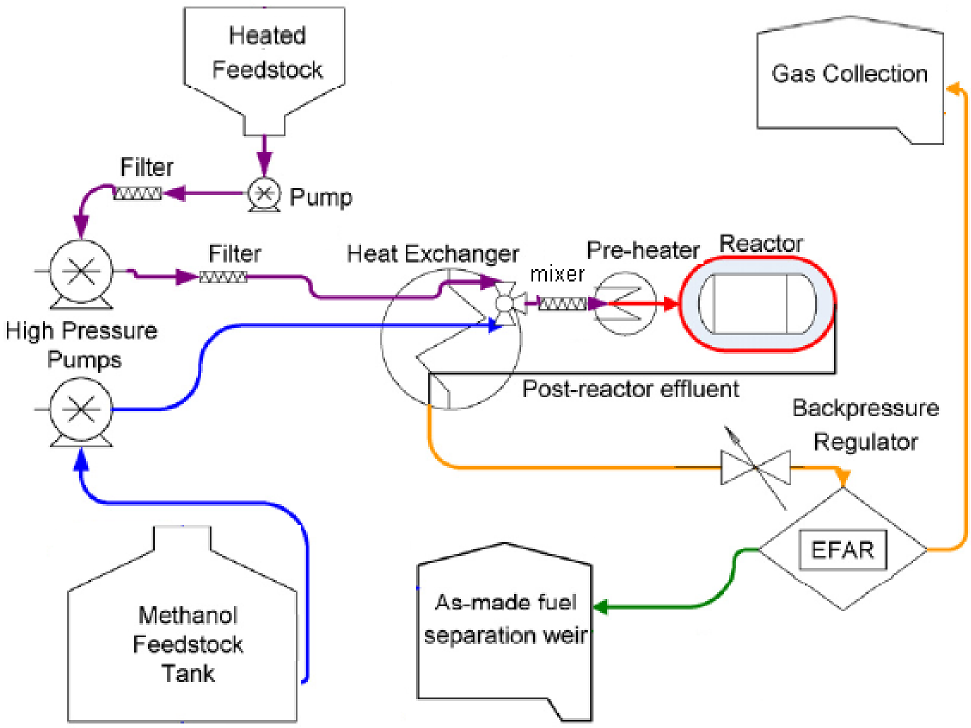


Fig. 17: *Mini-plant for continuous production of biodiesel for sub- and super-critical operations.*

The operating parameters at which the mini-plant was driven are gathered in Table 1:

Tab. 1: *Reaction conditions of the plant for continuous biodiesel production*

Reaction conditions	
Temperature range	250-400°C
Pressure range	150-250 bar
Trans-esterification reagent	Methanol/Ethanol/Ethyl Acetate
Type of oil	Waste cooking oil
Catalyst + Foam Support	The reactor could operate without catalyst with a static mixer mean (ceramic or metallic foam). The foam can host a stable and active catalyst.
Productivity	0.1 liter of FAME per minute

The mini-plant was erected at the end of 2012 and operated in 2013 for the production of biodiesel. Bio-derived ethanol was used as the transesterification agent, with the purpose of achieving a fully renewable biodiesel fuel (FAEE: fatty acid ethyl esters mixture). Starting from a reference condition of 300°C, 200 bar, alcohol-to-oil ratio of 42, which led to a FAEE yield of 11.9%, these parameters were varied to investigate their individual effect. The most relevant effect was given by the change in temperature, for which the FAEE yield reached 56.4% at 340 °C, the other conditions being the same. The pressure increase was also beneficial towards the oil conversion, since at 240 bar (at 300 °C and mole ratio of 42) the FAEE yield was 28.7%. As far as the mole ratio was concerned, it was found that its further increase above 42 was not leading to any improvement in the oil conversion. Finally, the impact of residence time in the reactor was also estimated, by operating the reactor without the internal pellet filling (i.e. resulting in a larger reactor volume), and the FAEE yield increased from 11.9% to 15.7%.

One has to underline that the obtained results are very encouraging because the pilot scale of this plant proved to be effective given the major issues of oil and alcohol homogenization, and, more importantly, in continuous mode. From the same set of experiments, it was found that significantly higher FAEE yields could be obtained for higher residence times, which could be the object of future experimentation.

The mini-plant is currently located at Politecnico di Torino, in Alessandria (AL), Italy (Figure 18), where it is available for research and external consultancy. Its flexibility and robustness encourages a profitable exploitation to explore the wide operating conditions for biodiesel production that this plant can afford. Moreover, the possibility of enforcing combined conditions of temperature of 380°C and pressures of 250 bar, further widens the window of processes that can be experimented inhere.

Fig. 18: *Mini-plant for biodiesel production, located at Politecnico di Torino in Alessandria, Italy (Source: POLITO).*

1.3.4 Case story: Biomass gasification in view of decentralised ammonia production

Introduction

Of the various types of alternative energy sources available, it is projected that bioenergy could sustainably contribute between a quarter and a third of global primary energy supply by 2050. Biomass which currently provides 10% of global primary energy demand is the sole form of renewable energy which can replace fossil fuels in all energy markets for the production of heat, electricity and commodity chemicals. Furthermore due to its almost closed carbon cycle, use of biomass as one of the main sources of primary energy production can significantly reduce global greenhouse gas emissions. Also available globally are low value waste feedstocks such as refuse derived fuel which in Europe alone represent 55% of non-recyclable waste. Thermochemical conversion of these low heating value feedstocks to a combustible gas by gasification at temperatures greater than 800 °C is recognised as the most efficient thermal conversion process as it can capture up to 80% of the energy in these materials. Moreover, the high biomass to energy conversion efficiencies obtained during gasification can be further enhanced through the use of combined cycles for generation of electricity.

Gasification of a wide variety of low heating value wastes including miscanthus, wood chip and refuse derived fuel for the production of electricity was carried out in a novel 2.0 MW up-down-side draft gasifier developed by ITI Energy and Newcastle University using air as the gasifying agent. On the basis of the results obtained from air gasification of these feedstocks, the development of a novel oxygasifier system for the production of ammonia was carried out under the CoPIRIDE project. Here, the focus was led on the quality of the syngas generated from this novel gasifier necessary for the production of ammonia and other commodity chemicals.

Another key aspect was the development of plasma reactors, suited for the conversion of the syngas to ammonia and other chemicals and of novel nanostructured microporous catalysts to be applied within such a reactor.

Hybrid integrated plant concept with 2nd Process Intensification field for ammonia production from biomass

In the framework of the CoPIRIDE Project it was aimed at to integrate several intensified unit operations and achieve an intensified chemical plant. Ammonia production was deliberately chosen since this is a very well-known and highly mature technology. Nevertheless, the number of unit operations is very large and energy inefficient. The integration of intensified unit operations cuts down these steps and creates energy and mass transfer efficiency, therefore making the technology sustainable since the feedstock is renewable (biomass or biomass waste). Due to the distributed nature of biomass feedstock, the ammonia plant must also be a distributive production platform and for sustainability, it must not carry the burden of 'economies of scale', a common character of the centralised feedstock (i.e. fossil fuels) and centralised production. Gasification system development is the first and most important step in an integrated intensified ammonia production. Gasification system includes oxygen powered gasification of biomass (OxyGasification), oxygen/nitrogen separation, cleaning of syngas generated from the gasifier reactor, hydrogen separation from syngas for use in ammonia synthesis and environmental safeguards including process water clean-up and recycle. Fig. 19 shows the developed novel gasification system by applying the principles of Process Intensification so as to achieve high energy conversion efficiency and quality syngas.

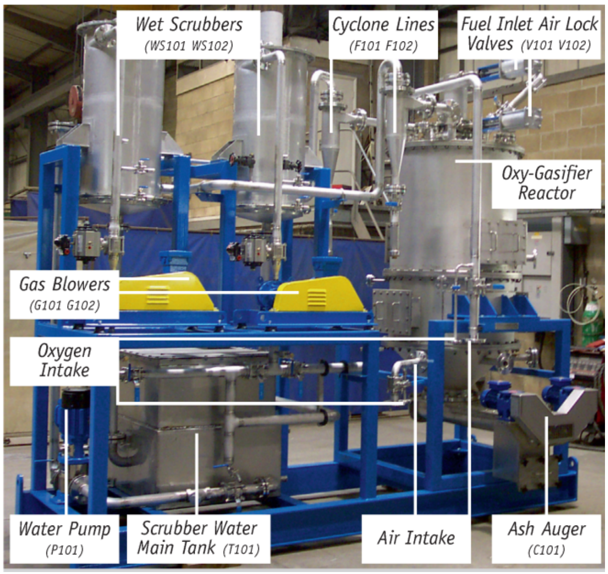


Fig. 19: *Intensified gasifier reactor (Source: ITI).*

Several syngas cleaning techniques have also been developed and tested for their efficiency and suitability. These intensified syngas cleaning techniques operating at high or low temperatures are essentially chemical reactors/mass transfer facilities in which more than one intensification field is present; similar to the other reactors developed for ammonia synthesis. Syngas cleaning techniques (simultaneous removal of tars, moisture and heavy metal ions) developed include the use of novel nanostructured microporous materials which are also used as microreactors or catalyst support. Polymeric versions of these materials are generically called PolyHIPE Polymers (PHPs) which reflects their method of preparation through High Internal Phase Emulsion (HIPE) polymerisation. The hierarchic pore structure of PHPs prevents diffusional restrictions and allows selective adsorption of surface active species in which the thermodynamic driving force is based the 'confinement phenomenon'. Fig. 20 illustrates the hierarchy of the pore structure of PolyHIPE Polymers.

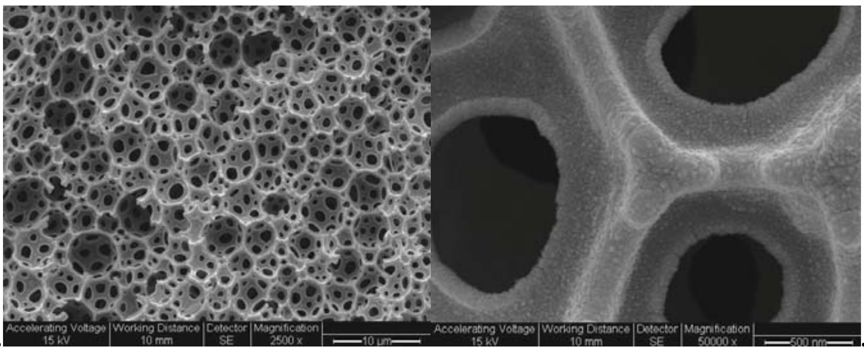


Fig. 20: *Hierarchic pore structure of PolyHIPE Polymers with increasing magnifications. Scale bar 10 (left) and 0.5 (right) micrometer (Source: University of Newcastle).*

The performance of these materials in syngas cleaning is illustrated in Fig. 21. Up to 98.5 % of the tar could be removed.

Fig. 21: *Flame colour before (left) and after (right) gas clean-up (Source: ITI).*

The University of Newcastle developed and investigated a catalytic discharge barrier reactor in three different electrode configurations. It was applied for the conversion of the cleaned syngas made from biomass to ammonia or chemicals which can be produced by using the Fischer-Tropsch synthesis.

As mentioned, another key aspect was the development of a novel nanostructured microporous catalyst which is on the one hand active and stable when inserted in the plasma reactor and on the other hand, does not negatively influences the plasma, i.e. which does not promote the recombination of the generated ions. Good results were achieved with coated Ni/SiO_2 catalysts as shown in Fig. 22 which exhibit a high dielectric constant.

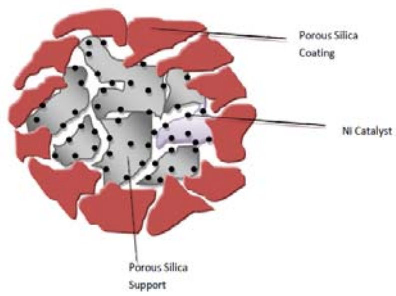


Fig. 22: *Coated catalysts for plasma intensification field (coating: material of high dielectric constant, core Ni/SiO_2 ; Source: University of Newcastle).*

Conversions per pass between 10 to 18 % were achieved at ambient pressure and relatively moderate temperatures up to 250 °C. For comparison, the conventional process with similar conversions requires pressures between 100 – 250 bar and temperatures between 350-550 °C.

The intellectual property of the developed technology was protected by patents, filed for the ammonia production by integrated intensified processes and for an integrated intensified biorefinery for gas-to-liquid conversion. Applications are seen in agriculture, i.e. in case of decentralised ammonia production by producing a novel kind of fertiliser, and in case of using the Fischer-Tropsch synthesis, the conversion of syngas from (waste) biomass to fuels and chemicals.

1.3.5 Novel manufacturing techniques & modular microreactors

In the past, microstructured reactors have successfully been applied in laboratories and in several cases even on the pilot and production scale for the intensification of chemical processes. Currently available techniques are well established to enable the commercial manufacture of laboratory reactors in small series and of pilot reactors. However, there was still a lack of appropriate and cost-efficient manufacturing techniques for microstructured devices which are suited for the high throughputs of the production scale. The development and improvement of such manufacturing techniques was a major goal within CoPIRIDE.

A typical design of micro- and millistructured reactors is a stack of alternating reaction plates and heat transfer plates each of them bearing microstructured channel arrays. Depending on the application, the channels can also be coated with a catalyst. Due to the high ratio of the heat transfer surface to the device volume, such reactors are capable of transferring much more heat than conventional reactors with a comparable reaction volume. This allows for better process control and for intensifying chemical processes, e.g. by using highly active catalysts and by operating in so-called Novel Process Windows, such as elevated temperature and pressure. Thus, the reaction time can be reduced from hours to minutes or even seconds.

Wet chemical etching or mechanical machining processes such as milling are still most frequently applied for microstructuring metal and especially stainless steel plates. Both techniques are comparably expensive, limited in size and less suited for an economic mass production. Within CoPIRIDE, IMM and its industrial partners, the companies Wetzel and Laserzentrum Schorcht, jointly developed novel manufacturing techniques for microstructured reactors. They are based on roll embossing and laser cutting for the fabrication of structured sheets. For the application of these techniques, a corresponding concept for modular stacked plate reactors was developed at IMM. The new design for the reactors is now a stack of alternating reaction layers and heat transfer layers (see Fig. 23). Each layer is built of a sheet structured by roll embossing, frame(s) around it and a flat separation sheet which also covers the microchannels.

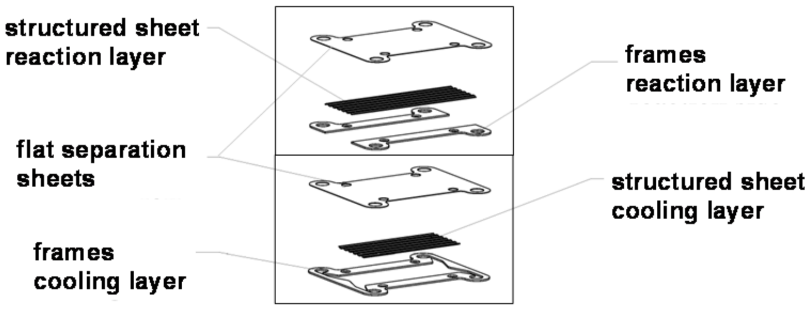


Fig. 23: *Basic design of a stacked plate microreactor in exploded view (Source: IMM).*

Fig. 24: *Modular, vacuum brazed microreactor. (Source: IMM)*

This novel reactor concept allows for adapting the reactors to a wide variety of different chemical reaction processes, but it also enables a cost efficient manufacture since this variety of reactors can be made by a small number of tools only, i.e. mating rolls for roll embossing. An example is that from the coiled microstructured strip, sheets of different length and shape can be separated, e.g. by laser cutting.

Fig. 25: *Assembly for roll embossing at Wetzel company (left) and downstream side of the mating roll with a microstructured strip of 60 mm width (right) (Source: Wetzel GmbH).*

They can be differently combined and treated, e.g. coated with a catalyst and joined by different techniques: Vacuum brazing (Fig. 26) enables the manufacture of large microstructured devices capable of withstanding higher pressures, whereas laser welding enables the joining of sheets which have already been coated with catalyst.

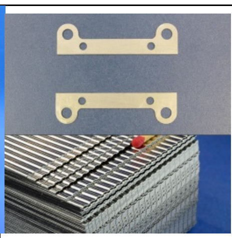
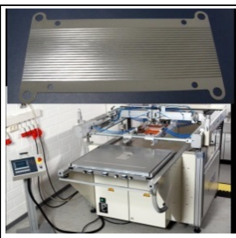
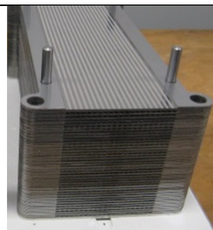
					
Roll embossing	Laser cutting of structured sheets, frames and flat separation sheets	Flattening of structured sheets	Coating of flat sheets with brazing paste using screen printing	Stacking of the device	Vacuum brazing and finishing

Fig. 26: *Manufacturing steps of vacuum brazed microreactors made of sheets structured by roll embossing (Source: IMM).*

As an additional variant type, foam reactors (Fig. 27) were developed where the microstructured reaction sheet was exchanged for a flat open-cell (metal) foam sheet (Fig. 28). These can be used to stabilize dispersions during multiphase reactions (e.g. liquid-liquid reactions) and can also be coated with a catalyst. This reactor type can be joined both by laser welding and vacuum brazing. Yet, the potential of these novel manufacturing techniques for cost reduction and resource efficiency holds for mass products produced at large scale.

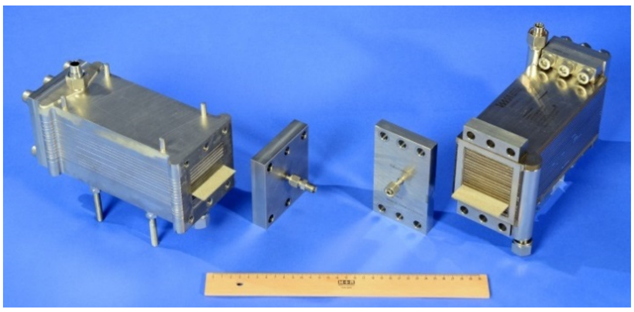


Fig. 27: *Vacuum brazed foam reactor for supercritical biodiesel production at up to 400 °C and 250 bar (left) and laser welded foam reactor (right) (Source: IMM).*

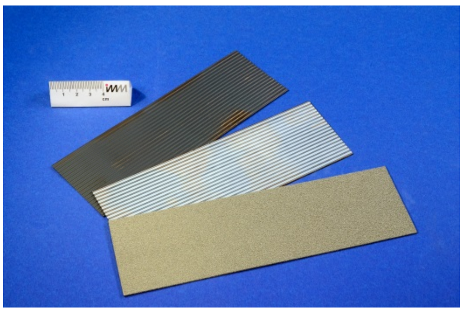


Fig. 28: *Different reactor sheets (roll embossed, uncoated and coated with catalyst, open-cell nickel foam sheet) (Source: IMM).*

Within CoPIRIDE, embossing rollers with slightly different channel shape were developed; one has a simple waved profile for joining the stacked plate devices by laser welding, and the other one exhibits additional depressions to be filled with brazing alloy in the case of joining the stacks by vacuum brazing. After the novel manufacturing techniques and all these types of reactors, based on sheets with 60 mm structured width, had been successfully developed, the next step was transferring the technology to larger dimensions. This was done by following two different approaches, which in future could also be used combined: First, by manufacturing a mating roll with the larger structured width of 150 mm, but producing microchannels of the same size and shape than the former ones with 60 mm structured width. This was realised for the microchannel structure suited for laser welded devices. Secondly, for larger vacuum brazed reactors, two sheets of 60 mm structured width were laid side by side, resulting in overall 120 mm structured width of one layer. The reaction sheets of the laser welded reactor (see Fig. 29) are made exchangeable which offers the chance to exchange them if the catalysts coated at the microchannel walls has to be renewed after a certain time of operation. Such an exchangeability of catalyst coated reaction plates in laser welded microreactors is an additional benefit of the developed novel manufacturing techniques. Besides several laser welded reaction modules (Fig. 29) in the large scale, also the first prototype of a large vacuum brazed reactor comprising reaction plates of the size (2x60) x 400 mm could be manufactured (Fig. 30).

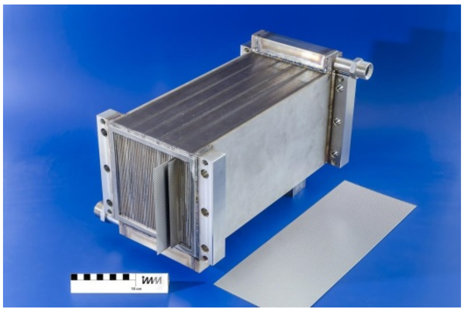


Fig. 29: *Large laser welded stacked plate microreactor, comprising exchangeable roll embossed reaction plates of the size 150 x 400 mm. (Source: IMM)*

Fig. 30: *Large stacked plate microreactor inside the vacuum furnace, comprising roll embossed reaction plates of the size (2x60) x 400 mm. (Source: IMM)*

Roll embossing is also a well suited technology for microstructuring other corrosion resistant and ductile metals like titanium or hastelloy. First vacuum brazed test reactors made of titanium had already been manufactured (Fig. 31).

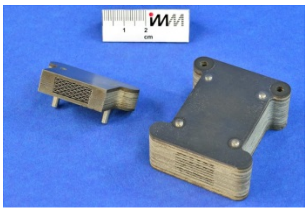


Fig. 31: *Test reactor made of titanium, vacuum brazed. (Source: IMM)*

Within the project the novel reactors have been intensively used and explored. A vacuum brazed foam reactor (Fig. 27) was applied for the production of biodiesel under supercritical conditions which are especially suited to use waste oil as feedstock. Both, vacuum brazed (Fig. 24) and laser welded microreactors comprising roll embossed sheets, had successfully been applied in chemical pilot plants for the epoxidation of soybean oil and the propoxylation of alcohols.

1.3.6 Exploration of Novel Process Windows

Novel process windows (NPW) are defined as chemical intensification by exploring p, T, c parameter settings not or hardly possible in conventional equipment (harsh chemistries). Main effect is the boosting of reactivity by a factor of 100 - 1000; yet, other effects, e.g. on selectivity, are also known. The definition further includes effects on a full process scale which reduces the number of equipment (process simplification) or enhances the functionality or performance of equipment (process integration), which are enabled by flow processing or more specifically by the chemical intensification given above. In the framework of the CoPIRIDE project, this takes the form of a search oriented to translate low T to high T processes, in order to achieve increased productivities due to faster reactions, and to shift batch synthesis into continuous (flow) synthesis, to gain the advantages of flow processing compared to batch. The three most relevant ones of the six case studies in the CoPIRIDE project are discussed below. They comprise NPW cases of Fig. 32 utilized for the living anionic polymerization, the soybean oil epoxidation and the supercritical biodiesel synthesis.

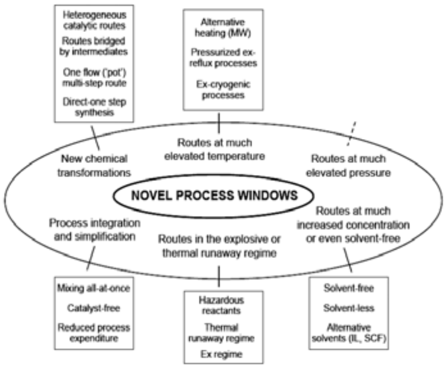


Fig. 32: *General scheme for NPW search (Hessel et al., Chem. Eng. Sci., 66(7), 1426-1448, (2011))*

Living anionic polymerization

The anionic polymerization case is an example of reaction driven study, where the main aspect followed was the understanding of a model reaction for further exploitation to related production. The novel process windows in this case are twofold. NPW 1 concerns the use of high temperatures for the reaction, NPW 2 concerns the flow pattern in a continuous tubular reactor, in this case high-viscosity flow with isolated, parallel streams, due to missing radial diffusion at higher monomer conversion. This is the radial analogue to 'axial' segmented flow. The reactor was investigated both with 2D and 3D simulations, where the latter show the best agreement with experimental results, although at the price of much higher computational effort. The model was developed for butadiene polymerization, using various correlations to determine the value of the physical parameters as a function of the polymer properties and of the polymer concentration, which does affect viscosity, diffusivity, heat capacity and heat transfer behavior of the reactive mixture. The predicted molecular weight and polydispersity index (PDI) where validated against experimental results obtained at USTUTT as shown in Fig. 33. This feasibility proof can be applied to other polymerizations, needing determination of the properties and conditions and driven by market analysis towards high added value specialty products. For this case study a working prototype (lab scale) was produced, able to polymerize different monomers with reliable and reproducible controlled outputs. Based on the

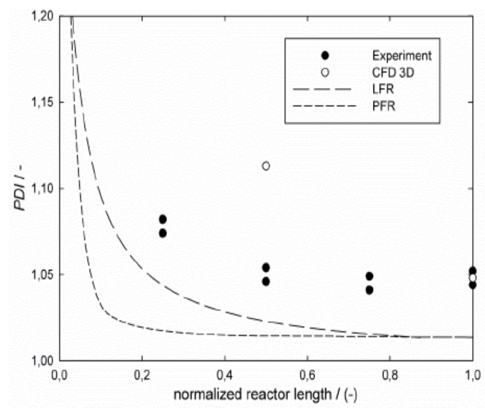


Fig. 33: *Comparison of PDI values at different capillary lengths obtained from modeling and experiment. (Source: S. Schulze et al., Green Process Synth 2013; 2; 381-395).*

results produced, and from theoretical evaluations, a full model of micro flow polymerization was developed that yields the values of all the relevant physico-chemical parameters at any point of the reactor.

Soybean oil epoxidation

The soybean oil epoxidation is an example of a process driven study, where a detailed model of the reaction is to be used in an advanced optimization algorithm in order to find an optimal novel process window. In this case three NPW issues are encountered. NPW 1 concerns the use of high temperatures for the reaction to increase the conversion rate; NPW 2 is about process simplification because of the use of a smaller excess of H_2O_2, which simplifies downstream purification of the products; NPW 3 concern safety increase in production. NPW 1 was initially investigated by means of simulations using simplified, lumped kinetics, and although promising results were found, a more precise model was needed for further steps. The latter model is also needed in order to exploit NPW 2. It was developed jointly by UNA and TUE, by means of kinetic experiments in multiple devices and by determination with imaging methods of the specific interface area, a_V, see *e.g.* Fig. 34. The determination of a_V allows for an explanation of the mechanistic effects of mass transfer on the product formation and degradation. The model was afterwards employed as a source for different semi-heuristic methods searching for optimal process designs, restricted by the needs provided by an industrial partner. The calculation stage yielded promising options and the procedure was standardized in a way that makes it re-applicable on any process of choice. However, the final numerical stability in the calculations proved not sufficient for its smooth deployment, due to the overall complexity of the system under study. This high level of complexity encountered at the moment prevents an easy transferability of the results toward other processes. Still, the work of MIC, IMM and UNA allowed the construction of a working pilot plant for this reaction, where notable improvements over the standard batch synthesis are obtained. The best approach obtained in the end is a mixed one, where a combination of a microreactor and a milli reactor with built-in static mixers takes charge of the first, fast and exothermic, stage of the process and act as the feed of a large CSTR where the "non-boostable" stage of the process is done. This approach demonstrated itself as the most cost effective.

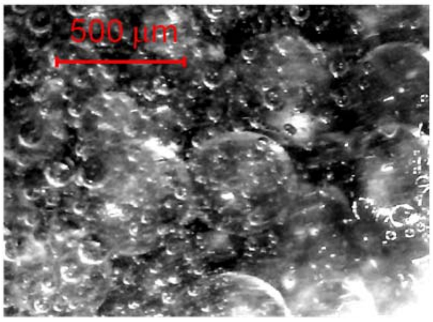


Fig. 34: *Imaging of droplets in the reaction environment for a sample batch run.*

Supercritical biodiesel synthesis

The biodiesel synthesis is an example of a life-cycle driven study. While the transesterification reaction is well known large issues related to the real environmental impact of biofuels remain open at the moment. For this reason, the LCA and SLCA methodology allowed to define the critical points to address, and to identify the most relevant parts of the process in terms of environmental impact. This in turns enabled a targeted study to achieve the highest improvement in terms of environmental footprint of the process. NPW issues encountered in this case study concern NPW 1, operation under supercritical conditions (high p, T) and NPW 2, performing the reaction along a new reaction path in order to form side-products with higher margin. NPW2 was after initial trials not followed further due too low activity for the reaction. However, this case is relevant for dissemination because the same methodology, *i.e.* using supercritical reactant conditions, can be applied to other similar products in different market segments. The most striking result of this case study is the design and construction of a pilot scale continuous supercritical plant able to run on a wide variety of different feedstock, ranging from virgin vegetable oil to waste frying fats at the Alessandria site of CTX.

Conclusion

The largest success of NPW was achieved for the living anionic polymerization and actually with an NPW which was outside sight when initially defining NPW. The greater initial promise in the high-T processing for the soybean oil epoxidation was hampered by effects unforeseen of all existing kinetic models, including that of UNA. Most likely a H_2O_2 destruction much larger than known sets a limit in the exploitation of that NPW. Still, an operation of about 30-40 ^{0}C higher was realised on a pilot scale with micro structured reactors which would have been impossible for conventional equipment. The interaction with the other applications was hampered by frequent changes in process options, limited use of NPWs in reality, and insufficient fundamentals at hand. In general it was realised that the chosen processes did miss many of the features, which the numerous processes have that have been successfully boosted by NPW on a laboratory scale. The multiphase processing of the soybean oil epoxidation, *e.g.*, added much complexity, not in favour of a NPW exploitation. Thus in a way it was real-life confrontation, at least for the processes considered, which hindered a more far-fetching exploitation of NPWs and in the same way of flow chemistry and micro process technology. Nonetheless, it is possible to draw some precise indications from the practical experience the project gave:

- It is of utmost importance to determine at the earliest stages which will be the kind of development to follow, *i.e.* will it be reaction, reactor or process driven. This is particularly important when risky innovations are attempted (i.e. high-T processing), where each step may prove challenging even when on the right track.
- Coupling the research stages from the early stage with SLCA is providing a guidance which may prove essential. This is particularly evident for the biodiesel case study, but was nonetheless essential for the epoxidation as well, where precise information on the environmental effect of the different reactants and leading to precise design choices.

1.3.7 Process development accompanying cost analysis and LCA

Increasing sustainability within the European chemical industry was a key objective of the CoPIRIDE project. In order to achieve this challenging goal, process development activities were accompanied by Life Cycle Assessment and Life Cycle Costing studies in order to provide decision support regarding the most sustainable option right from the start. This was done by virtue of the awareness that the degree of freedom is at highest at the beginning of process design and guiding directions for technology and process design choices prior to these developments is most effective. Starting from a broad screening of a wide range of alternative solution candidates for one given process design task, the complexity of evaluation was increased in parallel to the progress of development concentrating on the most environmentally benign and cost efficient concepts (see Fig. 35).

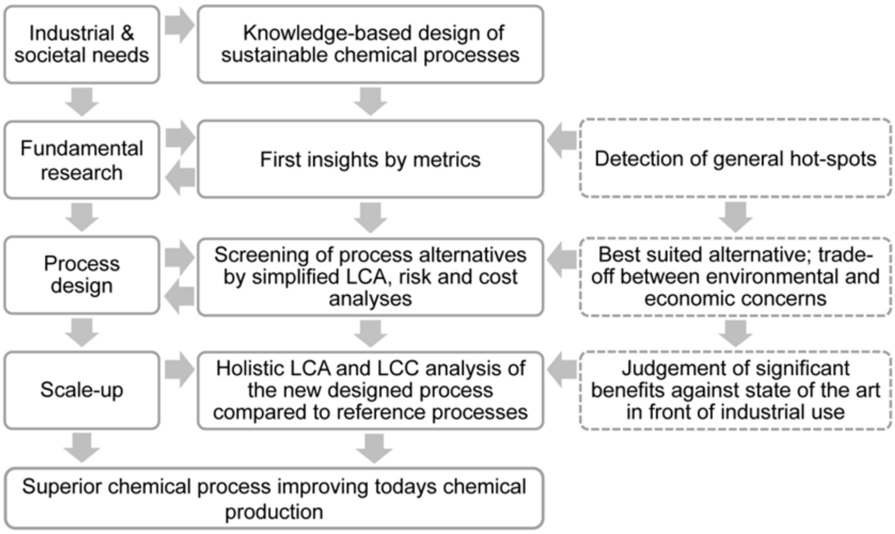


Fig. 35: *Evaluation approach followed within CoPIRIDE. Source: UNIJENA, D. Kralisch et al., Grenn Chem 2013, 15, 463-477.*

The promising results obtained by this internal sustainability controlling confirmed the benefits of such a holistic design approach. More sustainable processes were developed for chemical core processes such as the epoxidation as well as transesterification of vegetable oils, polymerisation and hydrogenation reactions.

A good case study to exemplify such multi criteria design tasks is the development of a novel biodiesel production process within CoPIRIDE. On the one hand, relevant environmental savings are requested from biofuels in the EU. Reaching this target is not granted in the light of necessary land use changes, significant emissions during farming, biodiversity issues and last but not least the competition to food. It highly depends on the feedstock utilized. On the other hand, biodiesel has today the highest market penetration of all biofuels developed within the last decades and process intensification strategies would have here the broadest impact on biofuel industry. But, increasing feedstock prizes result in lower earnings for European biodiesel producers. All in all, sustainable biofuel availability is not ensured yet. Instead, there is an urgent need for novel, intensified processing methods and robust concepts for environmentally benign & cost efficient processing taking into account the important feedstock impact on resulting saving potentials.

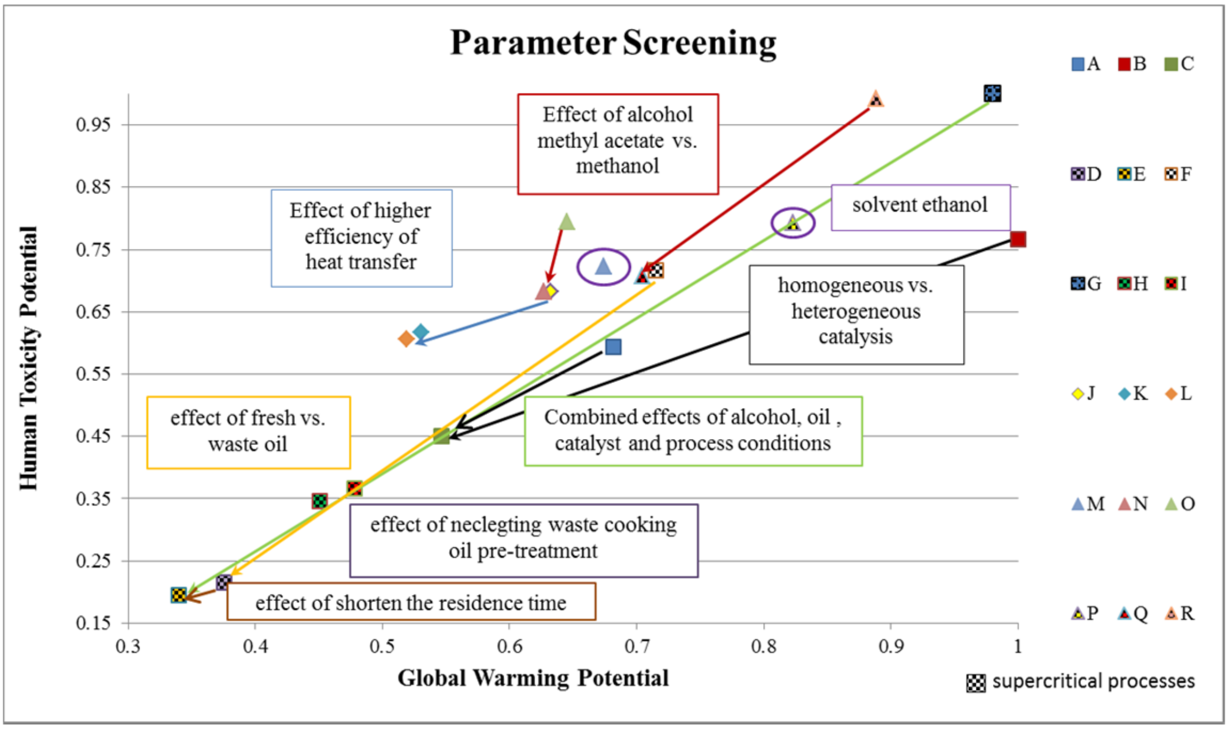


Fig. 36: *Parameter screening of various biodiesel production designs regarding the Life Cycle Impact Assessment criteria Human Toxicity Potential and Global Warming Potential (scaled effects) (Source: as Figure 35),*

The sustainability evaluation in this case study started with a screening of eighteen alternative process parameter configurations with regard to environmental impacts (see Fig. 36). Later in the design process, a full Life Cycle Assessment of the best case was coupled with an assessment of the future production costs and potential environmental, health and safety risks and compared with industrially established reference processing alternatives.

Now, the pilot plant developed within CoPIRIDE by the Department of Applied Science and Technology at the Politecnico di Torino in cooperation with CHEMTEX Italy is designed to utilize waste vegetable oils in a very efficient manner. This development allows an up to 70 % reduction of greenhouse gas emissions induced by biodiesel production compared, *e.g.,* to a conventional homogeneously acid catalyzed process utilizing fresh soybean oil. This means the avoidance of 0.86 t greenhouse gas equivalents referred to the production of 1 t biodiesel! Also, the overall production costs are drastically reduced compared to processes using fresh vegetable oil as feedstock. Last but not least, compared to conventional as well as supercritical processing in a continuous stirred tank reactor, the CoPIRIDE process performed in intensified flow reactors with a residence time of only a few minutes reduces environmental, health and safety risks arising in case of an accident.

By consequent coupling of process design and sustainability assessment an intensified continuous flow biodiesel production from waste concept has been developed with a high likeliness of widespread transfer in the European biofuel industry.

1.4 Potential impact, main dissemination activities and exploitation of results

1.4.1 Potential impact

Introduction

The CoPIRIDE project proposal was a response to the call for projects "NMP-2008-3.2-1 Implementation of process intensification strategies in industrial scale" within the EU FP7 Programme 2008, Cooperation, Theme 4 – Nanosciences, nanotechnologies, materials and new production technologies (NMP). This call was part of the subactivity "Adaptive production systems" in the activity "New production". The programme text is setting the scene also with regard to expected project impact. Under "New production" it is stated that "A new approach to manufacturing is required for the transformation of EU industry from a resource intensive to a sustainable knowledge-based, eco-innovative industrial environment … ." This is developed further for the subactivity as follows: "The key objective is to develop production systems and elements for knowledge-based factories through holistic manufacturing engineering concepts. The systems should automatically and continuously adapt production resources and processes in an optimal way with respect to business and production objectives as well as market and technical conditions. … The scope includes discrete manufacturing and process industries, supporting also the trend towards miniaturisation, as well as construction." In the specific topic (NMP-2008-3.2-1) finally the following expected project impact is specified: "European based chemical production meeting the challenges of increased product diversification, substantially shorter time to process/market and flexible production capacity in accordance with product and market development. A substantial drop in capital expenditure for new plant and/or for retrofit of high–performance intensified devices into existing infrastructure for the high value-added product market … ."

(Potential) Impacts of project and of its achievements

The European chemical market is under considerable cost and ecological pressure, especially in view of the new emerging markets and production capabilities in Asia. Process Intensification (PI) is seen as promising concept to increase competitiveness and sustainability of chemical processes CoPIRIDE has followed the concept of PI with a special focus on exploring Novel Process Windows (NPW) not accessible with conventional reactor technology and has targeted to provide entirely new concepts – both for process steps and equipment/plants.

New modular plant concepts & production concepts

The concept of the **Evotrainer** of Evonik Industries AG as **flexible and mobile plant in container format for small-scale production** covering the whole manufacturing chain to the chemical product in one system was significantly brought forward and fostered in the project as not only documented by the development, realisation and operation of two Evotrainers for the piloting of the synthesis of a special polymer in a two-step synthesis process. It is aimed to develop the process to full maturity therein under real-life production conditions before building a large-scale facility. The advantage of working in the Evotrainer here with the same kind of equipment and processes as those found in industrial scale facilities makes the potential transfer to a large-scale facility later on easier, cheaper, faster and more secure. Another important aspect of the Evotrainer concept is that the Evotrainer can be used as central infrastructure covering lab development, piloting and production. This approach can shorten development time from product idea to the final production process significantly (**reduction of time-to-market**). Furthermore, the Evotrainer approach foresees concepts for an **easy adjustment of production capacity closely to market trends** without elaborate retrofitting efforts. Through its **mobility** a geographical following of market trends is feasible too. Also **new business models** like “rent a plant” are proposed. The Evotrainer concept therewith bears the potential of **raising competitiveness of speciality chemical production** in Europe. Also the second plant concept developed in the project, the Flow Miniplant concept of the **SME** Microinnova Engineering GmbH, addresses comparable challenges and delivers corresponding solutions whereby a stronger focus is given to **integration possibility into existing facility infrastructures** (e.g. larger fume hoods), the use of microstructured reactors as a central element and **modularity** of the small-scale production unit. The Flow Miniplant concept already faced its first successful commercial implementation shortly after project end. Promotional activities are underway for a broader uptake. Finally, the activities around the biomass gasification target at a **decentralised production** of ammonia or other chemicals.

New modular microreactor concepts and manufacturing techniques

Microreactors are central enabling tools for PI and NPW. The project **fostered** this **innovative technology** and therewith its **broader uptake and exploitation** by the development of new modular reactor concepts and the opening of more cost efficient fabrication approaches especially in view of large-scale format reactors based on mass-manufacturing suited fabrication techniques. The developed modular microreactors proved as **adjustable, flexible and high-performance equipment** (see epoxidation, propoxylation reaction) and a central element for the new plant concepts (see integration in the Flow Miniplant).

Process Intensification (PI) and Novel Process Windows (NPW)

In the project the deliberately exploration of harsh or otherwise very unusual process conditions for **PI (NPW) was followed in the chemical case studies**. The focus was especially on translating low temperature to high temperature processes in order to achieve **increased productivities** due to faster reactions, and to **shift batch synthesis into continuous (flow) synthesis** to gain the advantages of flow processing compared to batch. NPW were successfully exploited in the anionic polymerisation (**high product quality** reached), the epoxidation of soybean oil (**increased space-time yield**) and the supercritical biodiesel production (**opening processing of low-value feedstock** i.e. waste oils). The quite systematic effort within CoPIRIDE to confront all chemical case studies with NPW thinking led also to the generic learning a) of doing this as early as possible and defining precisely which kind of development to follow, i.e. reaction, reactor or process driven and b) to couple this also early with simplified LCA and ex-ante cost analysis to support development direction choice in order to reach a **full exploitation of this innovative concept (PI & NPW) and micro process technologies as linked enabling tool**.

Development accompanying cost analysis and LCA

Increasing sustainability within the European chemical industry was a key objective. So, **process development activities were accompanied by Life Cycle Assessment and Life Cycle Costing studies** in order to provide decision support regarding the most sustainable and efficient option right from the start. Starting from a broad screening of a wide range of alternative solution candidates for one given process design task (by simplified LCA and ex-ante cost analysis), the complexity of evaluation was increased in parallel to the progress of development concentrating on the most environmentally benign and cost efficient concepts. The systematic implementation of this internal sustainability controlling approach is **quite unique to CoPIRIDE** and led to promising results confirming the benefits of **such a holistic design approach which is suited for a broader uptake for promoting economic viable and sustainable processes**. In CoPIRIDE specifically, **more sustainable**

processes were developed for chemical core processes such as the epoxidation as well as transesterification of vegetable oils, polymerisation and hydrogenation reactions.

European dimension

The frame of the EU project allowed bringing **multi-disciplinary** expertise in **universities, research centers and enterprises** from different EU member states together in a **complimentary** manner to tackle the addressed challenges successfully and fostering **EU wide collaboration**. Both scientific and industrial partner benefited through the **uptake of scientific achievements into business** and the increase in interdisciplinary interaction to foster the generation of new knowledge. The project is so also contributing to the **transition to a knowledge-intensive European industry**. Especially the new plants concepts contribute to the move towards **adaptive production** in Europe. The results of the project – as outlined - will foster especially the **competitiveness of European chemical industry including SME** and contributes to **more sustainable European chemical production**.

1.4.2 Main dissemination activities

Project contents and results have been disseminated by the project partners by **more than 250 activities.** An insight in the main activities is provided in the following.

Naturally the main dissemination routes from academics and the R&D institutes in the project have been to publish project results in peer-reviewed journals and to contribute to scientific conferences. Remarkably, there have been a good range of central articles and contributions including also the industrial partners and thereby documenting the application orientation of the performed research and development works in the project.

Overview and main journal contributions

The project partners reported (see Table A1 in Chapter **Fehler! Verweisquelle konnte nicht gefunden werden.** for the complete list) **44 publications** linked to project works **in peer reviewed journals** for the reporting period. Two more are submitted. Furthermore, two diploma theses and one book chapter is reported. Among the publications, there are several joint publications from project partners addressing main project topics and documenting the good interaction in the project. Good examples with regard to this are especially the publications the following publications dealing with cost analyses and life cycle assessments of the main chemical processes considered in the project:

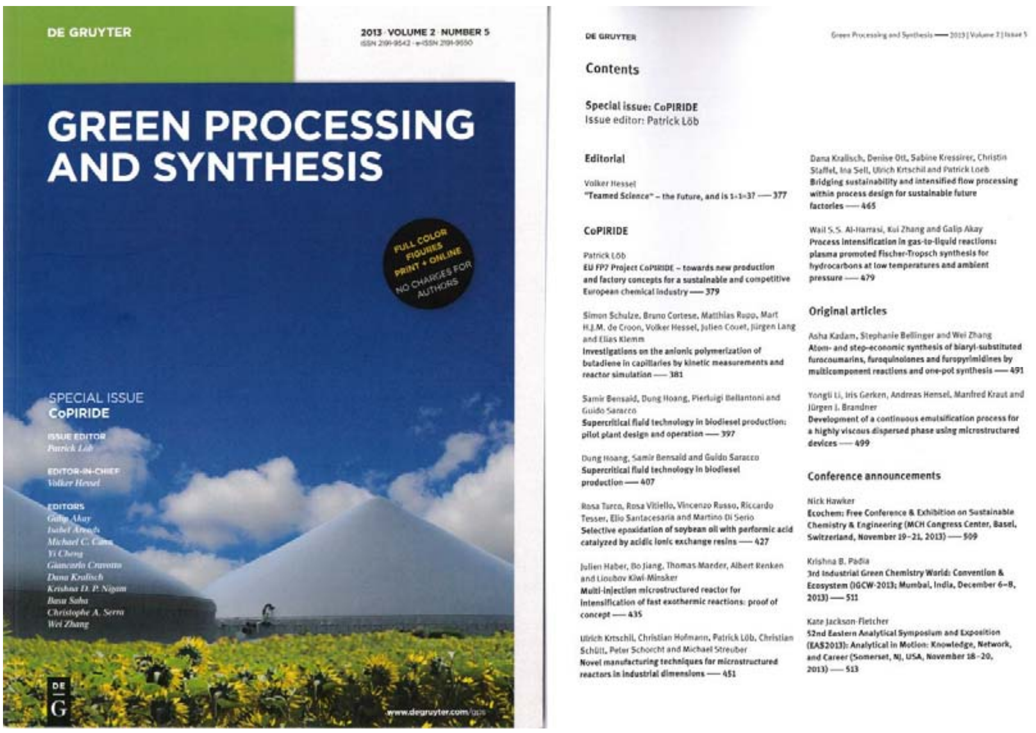
- D. Kralisch, I. Streckmann, U. Krtschil, E. Santacesaria, M. Di Serio, V. Russo, L. de Carlo, W. Linhart, E. Christian, B. Cortese, M.H.J.M. de Croon, V. Hessel,
  "Transfer of the epoxidation of soybean oil from batch to flow chemistry: guided by cost and environmental issues",
  ChemSusChem, 2012, 5, 300 – 311
- D. Kralisch, C. Staffel, D. Ott , S. Bensaid, G. Saracco , P. Bellantoni, P. Loeb,
  "Process design accompanying Life Cycle Management and Risk Analysis as decision support tool for green biodiesel production",
  Green Chem., 2013, 15, 463-477.
- S. Kressirer, L. N. Protasova, M.H.J.M. de Croon, V. Hessel, D. Kralisch,
  "Removal and renewal of catalytic coatings from lab- and pilot-scale microreactors,

accompanied by life cycle assessment and cost analysis”,
Green Chem., 2012, 14, 3034 – 3046.

- D. Kralisch, D. Ott, S. Kressirer, C. Staffel, I. Sell, U. Krtschil, P. Loeb,
“Bridging sustainability and intensified flow processing within process design for sustainable future factories”,
Green Processing and Synthesis, 2013, 2 (5), 465 –478.

The articles have been published in a range of journals to reach the targeted audience. Among the main journals used are: Green Processing and Synthesis (12), Industrial & Engineering Chemistry Research (6), Chemical Engineering & Processing (4), Catalysis Today (3), ChemSusChem and others (2). The journals used for publishing CoPIRDE results with the highest **impact factors** in 2012 were ChemSusChem (**7.475**), Green Chemistry (6.32), Chemical Engineering Journal (3.473), Fuel (3.375), Catalysis Today (2.98), Fuel Processing Technology (2.816), Topics in Catalysis (2.608) and Industrial & Engineering Chemistry Research (2.145).

A special highlight represents the special issue of “Green Processing and Synthesis” (5/2013) (see Fig. 37; http://www.degruyter.com/view/j/gps.2013.2.issue-5/issue-files/gps.2013.2.issue-5.xml) dedicated to the CoPIRIDE project results. This has been enabled by the co-coordinator V. Hessel being at the same time editor-in-chief of the journal. The coordinator P. Löb acted as issue editor. Besides the guest editorial by P. Löb, the project partners provided 8 articles to this special issue providing therewith a good insight in the central aspects of the project. To the guest editorial and three more articles open access is given.



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Special issue: CoPIRIDE
Issue editor: Patrick Löb

Editorial

CoPIRIDE

Original articles

Conference announcements

Fig. 37: *Cover and content page of the Special Issue of Green Processing and Synthesis dedicated to CoPIRIDE (http://www.degruyter.com/view/j/gps).*

Overview and main conference contribution

Project results have been reported in **about 130 oral presentations** including several keynotes and invited lectures. Supplemented has this been by **about 31 poster** contributions.

The contributions have been given in a range of different conferences to reach the targeted audience. Among them are the following:

- CHISA 2010 and 2012
- IMRET 11 (2010) and 12 (2012)
- WCCE 9 (2013)
- ECCE 8 (2011) and 9 (2013)
- ISCRE 22 (2012)
- EPIC 2011
- CAMURE 2011
- ACS Spring Meeting 2011
- AIChE Annual Meeting 2010
- CatBior2011 and 2013
- ProcessNet 2010
- Flow Chemistry Europe 2013

Joint activities with other EU FP7 projects

There have been a range of interactions and joint activities with other in parallel running EU FP7 projects. The highlights from these activities are two conference sessions during CHISA conferences dedicated to EU FP7 projects and a joint booth during ACHEMA2012 fair.

The **first joint session of EU FP7 projects** (F^3 Factory, CoPIRIDE, and PILLS) took place **during the CHISA 2010 conference** (19^{th} International Congress of Chemical and Process Engineering) in Prague (28.08.-01.09.10) with the following support from the CoPIRIDE project:

- Keynote lecture by *V. Hessel, U. Krtschil, G. Menges (IMM, TUE): CoPIRIDE - combining process intensification-driven manufacture of microstructured reactors and process design regarding to industrial dimensions and environment
- Oral presentation by *E. Santacesaria, M. Di Serio, R. Tesser, R. Turco, V. Russo (UNA): Epoxidation of soybean oil, a study on the possibilities of Process intensification.
- Oral presentation by *D. Kralisch, D. Ott, I. Streckmann, V. Hessel, U. Krtschil, G. Menges, R. Haidar (UNI JENA, IMM): Cost and Life Cycle Assessment –Accompanying Guidance during Process Design within COPIRIDE.
- Oral presentation by *J. Lang, R Schütte, H. Richert, M. Schwarz (EVI): New Plant Concept for Modern Chemical Factories with Intensified Processing – Least-Cost-Investment Plant EvoTrainer.
- Oral presentation by *G. Akay (UNEW): Process intensification fields, catalysts, hybrid reactors and intensified plants.

Fig. 38: *Impressions from CHISA2010 with CoPIRIDE participation. Left: session room. Right: CoPIRIDE booth. (Source: IMM)*

The **second joint session of EU FP7 projects** (F^3 Factory, CoPIRIDE, POLYCAT, SYNFLOW and PILLS) took place **during the CHISA 2012 conference** in Prague (25.-29.08.12) with the following support from the CoPIRIDE project:

- Keynote lecture by *P. Löb, U. Krtschil, V. Hessel, J. Lang (IMM, TUE, EVI): Combining process intensification-driven manufacture of microstructured reactors and process design regarding to industrial dimensions and environment - update on the results of the EU FP7 Project CoPIRIDE
- Oral presentation by D. Kralisch, S. Kressirer, *D. Ott, C. Staffel, U. Krtschil, V. Hessel, P. Löb (UNI JENA, IMM, TUE): Coupling of chemical process design and intensification with sustainability issues – Experiential report from the CoPIRIDE project.
- Oral presentation by *V. Hessel, L. Di Carlo, E. Klemm, D. Kralisch, J. Lang, W. Linhart, P. Löb, E. Santacesaria (TUE, MYT. USTUTT, UNI JENA, EVI, MIC, IMM, UNA): Novel process windows in COPIRIDE and beyond – from reaction intensification and new reaction design towards new process design.

This special session had an audience of about 60 people including the PTA of the project Keith Simons.

CoPIRIDE was represented also at the **ACHEMA 2012 fair** (18.-22.06.12) at the **joint booth of the EU FP7 projects** F^3 Factory, CoPIRIDE, PILLS, POLYCAT and SYNCLEAN (see Fig. 39 and Fig. 40) organised by DECHEMA. CoPIRIDE contributed via two project posters, handouts (newsletters, print out of the posters), a video provided by Evonik for repeated presentation on a monitor and via personal presence during the fair.

Fig. 39: *Joint booth of diverse FP7 EU projects at ACHEMA2012 fair with CoPIRIDE participation.*

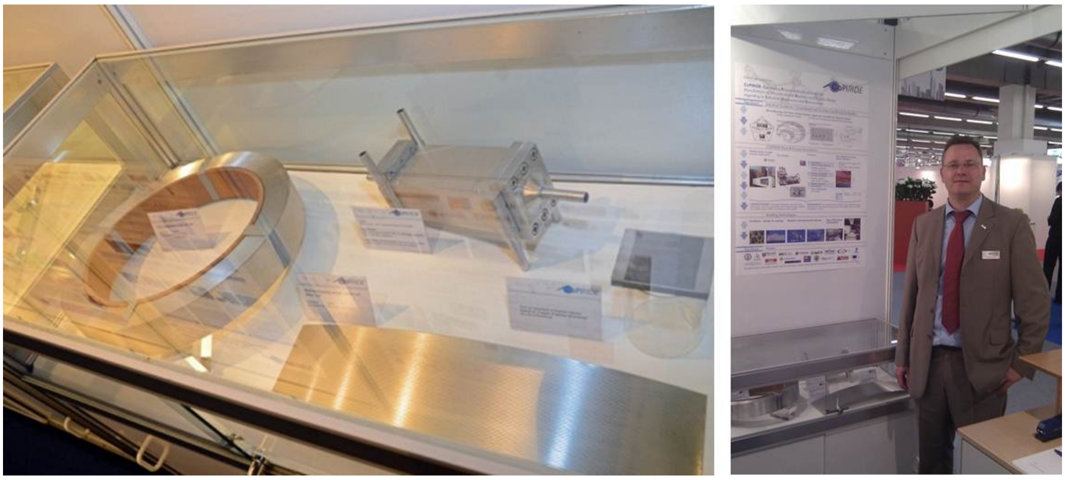

Fig. 40: *Details of the exhibits from the CoPIRIDE project on the joint booth of diverse FP7 EU projects at ACHEMA2012 fair: one focus have the new developed reactor manufacturing techniques and the reactor concepts.*

Public Dissemination Event

In order to disseminate central project contents and results in a concentrated form, a **Public Dissemination Event** has been organised. It took place as one day event subsequent to the 8th General Assembly held in Hanau/Germany at Evonik site on 22nd February 2013 for interested participants invited by the project partners and the co-ordinator (see Fig. 41). Approximately 70 participants from academia and industry, amongst others from BASF, Bayer Technology Services, Boehringer Ingelheim, Merck and Zeton, listened to the ten oral presentations and discussed new potentials offered by promising project results. Complementary, eleven posters and several exhibits were shown, enabling a comprehensive view on the project results achieved so far. Furthermore, tours to the Evotrainer were offered.

Fig. 41: *Impressions from the CoPIRIDE Public Dissemination Event, 22.02.2013.*

Other important dissemination activities

- Public information about the CoPIRIDE project is accessible via the **public web site** www.copiride.eu which will be kept in operation over the next few years.
- The published **Newsletters** as well as further information, e.g. a **flyer** about the developed new manufacturing techniques of microreactors and about the **Special Issue** of "Green Processing and Synthesis" dedicated to CoPIRIDE can be found under "News" http://www.copiride.eu/news.html of the public web site.
- The European Commission published a **Success Story** about the CoPIRIDE and F3 projects entitled "Modular, flexible, sustainable: the future of chemical manufacturing" on the Horizon 2020 web site (see http://ec.europa.eu/programmes/horizon2020/en/news/modular-flexible-sustainable-future-chemical-manufacturing)
- **Evonik** reported in a **series of three articles** in different publications of Evonik about the Evotrainer concept and its linkage to the CoPIRIDE project:
  1. in the quarterly science newsletter "elements", issue 4/2011, pp. 12-17, see http://corporate.evonik.de/sites/dc/Downloadcenter/Evonik/Global/en/Magazines/elements/elements-37.pdf
  2. in the publicly distributed Evonik Magazine, issue 2/2012, pp. 52-56, see http://corporate.evonik.de/sites/dc/Downloadcenter/Evonik/Global/en/Magazines/Evonik-Magazine/evonik-magazine-2-2012.pdf
  3. as title story in Evonik's employee magazine "Folio", issue 7-8/2013.

1.4.3 Training and education in academic teaching

It is worth mentioning that the above mentioned dissemination activities, there have been also two main activities dedicated to the topic of training and education in academic teaching: the organisation of a summer school for students from the CoPIRIDE project partner consortium and the preparation of four training courses around CoPIRIDE topics for use also outside the project consortium.

Summer school

On October 5, 2010, students, professors and other senior researchers as well as some industry representatives participating in the COPIRIDE project met on the Mediterranean island of Ischia, to get up to date for the latest progress of the other groups involved and discuss their newest results.

The Summer School took place in the NH Hotel, situated in the center of Ischia Porto. It started with the *Senior Researchers session*, which gave an overview over aims and topics in CoPIRIDE.

First, Prof. V. Hessel /TUE) gave an introduction to the concept of Novel Process Windows, followed by the presentation of Prof. E. Klemm (USTUTT), who showed how this concept can be applied using heterogeneous catalysts. Dr. D. Kralisch (UNI JENA) introduced Life Cycle Assessment as a tool to characterize a process not only via economic aspects, but to make it comparable to other processes considering social and ecological factors as well. U. Krtschil (IMM) presented a new approach for the cost analysis of microreactors. This was followed by Dr. J. Lang (EVI), who presented the Evonik Container Plant Technology concept. The senior session concluded with the presentation of the E.C. Officer Dr. S. Bøwadt, who showed the importance of interdisciplinary focus projects like CoPIRIDE from the EU commission's point of view.

Fig. 42: *Participants of the CoPIRIDE Summer School.*

The first presentation in the *Junior Scientists session* was given by B. Cortese (TUE). He showed the results of his parametric studies via numerical modeling of both the epoxidation reaction and the biodiesel formation. These two reactions were also investigated by other groups involved in CoPIRIDE: S. Hübschmann (UNI JENA) presented a life cycle assessment of the epoxidation reaction, and the first experimental results on this reaction were contributed by R. Turco and V. Russo (UNA). The experimental part of the biodiesel process was investigated by M. Chiappero (POLITO). Further presentations were given by S. Schulze (USTUTT) concerning a batch-to-conti approach in microreactors for anionic polymerization reactions, by V. Sifontes (AAU), who gave an introduction to his works on sugar hydrogenation over heterogeneous catalysts, and of A. El-Naggar (UNEW), who presented his works on membrane reactors for hydrogen generation from biomass.

The Summer School was a good opportunity for getting better to know the peers working on this project which afterwards facilitated co-operation.

Training courses

Four training courses have been prepared based on project works and results for use in public training and in academic teaching. It has been the ambition of the project to promote the proper utilization of the gained knowledge for future similar applications. For this, common lecture material has been prepared by the involved project partners. Around the industrial application examples, aspects like the determination of the intensification potential of the process, defining the aims of further process development, the decision for applicable tools to reach these aims and finally joining these activities in the creation and operation of pilot-scale reaction plants are covered. So, three of the four training courses are dedicated to the industrial application examples anionic polymerisation of butadiene, biodiesel production and epoxidation of vegetable oils. The following topics are thereby addressed: the chemistry behind the application, chemical technology: state of the art, identifying Novel Process Windows, intensified, flow through suited process protocol, estimation of the Process Intensification factor, pilot plan and Life Cycle Assessment. The fourth course gives an introduction into the topic of Life Cycle Assessment. The slides of these courses are available on request via the CoPIRIDE public website.

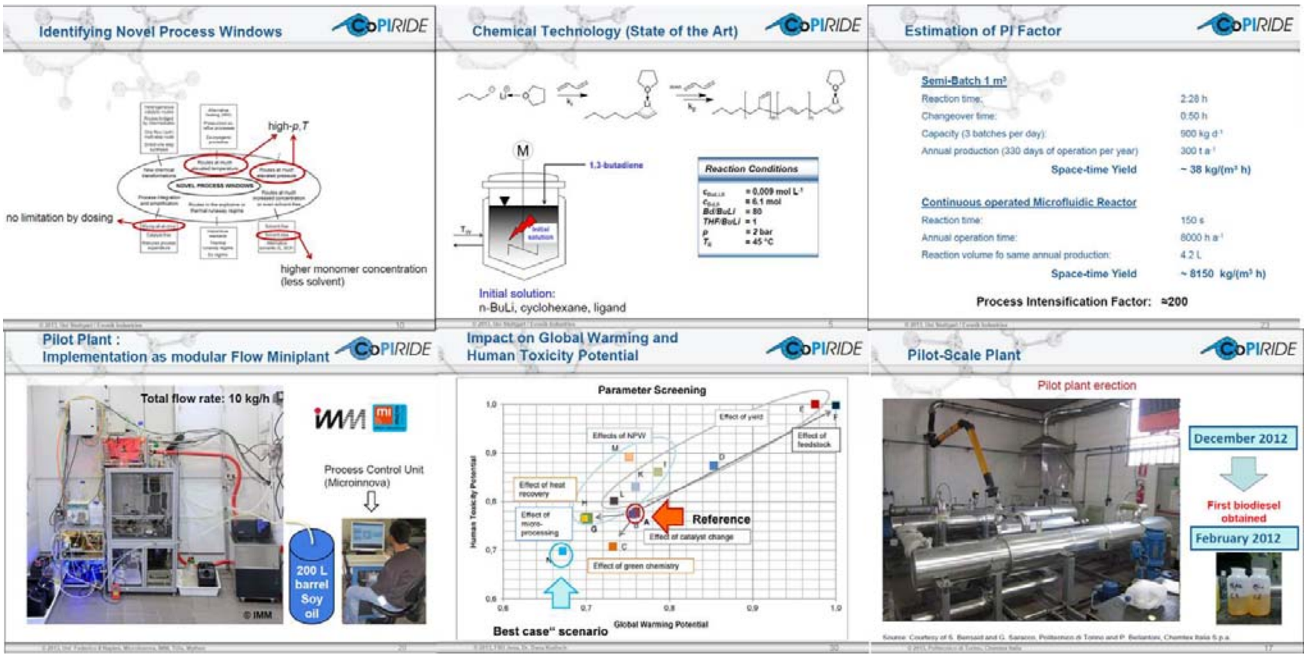

Fig. 43: *Example slides from the training courses prepared in the CoPIRIDE project.*

1.4.4 Exploitation of results

This chapter lists in view of commercial potential main project results and describes main activities of the project consortium to foster later exploitation / commercialization of them. Rather an insight than a complete summary is given due to confidentiality reasons and sensitivity of commercialization plans. First an overview regarding generated patent applications and from the partners identified main exploitable results is given. This is then followed by a description of main promotional activities.

As mentioned above in describing main objectives (see Chapter 1.2), CoPIRIDE has strived for improving technology at the catalyst – (reactor) fabrication – reactor – plant – process level up to a production stage. The following descriptions in this chapter document quite nicely that all topics have been addressed and for all of them exploitable results have been generated.

Patents

Project works and results lead to **6 patent applications**. Two of them are not published so far and so details have to be kept confidential; these two patent applications are relate to **novel process options** for the polymerization case study of Evonik Industries AG. Institut für Mikrotechnik Mainz GmbH filed two patents protecting **CoPIRIDE microreactor concepts and their fabrication**. Finally, University of Newcastle also applied for two patents around **ammonia production by integrated intensified processes** starting from syngas generated by biomass gasification and using a **novel catalyst system** and special membrane reactors for syngas cleaning. More details of the published patents are given in the following table.

Tab. 2: *List and details of published patents / patent applications resulting from CoPIRIDE project works.*

Type of IP Rights:	Embargo date	Application reference	Subject or title of application	Applicant
Patent appl.	01/03/2012	WO2012/025767	Ammonia production by integrated intensified processes	UNEW
Patent appl.	25/07/2013	WO2013/108047	Integrated intensified biorefinery for gas-to-liquid conversion	UNEW
Patent	21/03/2013	DE102012204178 B3	Mikrostrukturbauteil und Verfahren zu dessen Herstellung https://register.dpma.de/DPMAregister/pat/PatSchrifteneinsicht?docId=DE102012204178B3	IMM
Patent appl.	19/09/2013	WO2013EP55377	Microstructure component and method for the production thereof http://worldwide.espacenet.com/publicationDetails/biblio?DB=EPODOC&II=1&ND=4&adjacent=true&locale=en_EP&FT=D&date=20130919&CC=WO&NR=2013135866A1&KC=A1	IMM

Exploitable project results

The topic of exploitation of project results have been continuously followed and discussed during the complete project runtime e.g. by a first exploitation plan already in the proposal stage, by discussions during the Exploitation Strategy Seminar held in March 2011 and by consideration at the end of the project. It the latter case, the project partners specified a list of exploitable project results whereby an exploitable result is defined as an outcome of the project that meets two conditions: i) it has commercial relevance and ii) it can be commercialized as a stand-alone result. Besides the general advancement of knowledge generated in general especially by universities and research organizations, this list contains the following items:

- Plant concept in container-format for fine chemistry / pharmaceuticals productions, i.e. Evotrainer (Evonik Industries AG). Exploitation e.g. via licensing of container design.
- Erected mini-plant for biodiesel production as basis for research & consulting services (Politecnico di Torino).
- Pilot- and production scale microreactors for fine chemistry as basis for research & consulting services but also exploitation via licensing to an equipment manufacturer feasible. This exploitable result is strengthened by the corresponding patent applications. (Institut für Mikrotechnik Mainz GmbH).
- Modular plants for small to medium production scale for fine chemicals and pharmaceuticals, i.e. Flow Mini-Plant (Microinnova Engineering GmbH).
- Engineering services for biodiesel plants including novel processing route (Chemtex Italia, Politecnico di Torino).
- Laserwelding of micro heat exchangers and microreactors made of roll embossed plates (Laserzentrum Schorcht GmbH)
- New technology for ammonia synthesis and liquid fuel synthesis (University of Newcastle, ITI Energy Ltd).

Promotion of project results by promotional flyers and participation in exhibitions

Albeit not providing a complete picture, the commercial potential of project results is unveiled by promotional activities of the project partners in form of the preparation and distribution of flyers and the participation in exhibitions.

So, especially the project partners Laserzentrum Schorcht GmbH (laserwelding of stacks of microstructured plates), Institut für Mikrotechnik Mainz GmbH (novel modular reactors), Wetzel GmbH (microstructuring by roll embossing) and Microinnova Engineering GmbH (flow miniplant) have been actively advertising project results via flyers and via exhibits from the project at their booths on trade fairs. Examples are given in the following figures.

Fig. 44: *Flyer of Microinnova promoting project results towards potential customers..*

Fig. 45: *IMM booths during ACHEMA2012 and ACHEMASIA2013 with CoPIRIDE exhibits.*

Fig. 46: *Extract from IMM newsletter dedicated to ACHEMA2012 promoting CoPIRIDE reactor concepts.*

1.5 Contact details

1.5.1 Project coordination

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1.5.2 Project consortium

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