Integrated Process Control – SPIRE Projects with High Impact for Process Industry

Frans Muller (ProPAT, Leeds University, UK)
Manuel Pereira Remelhe (CONSENS, Bayer AG, DE)
Contents

- Sustainable Process Industry through Resource and Energy Efficiency

- Case Studies & Technologies
- Expected Impact
- Adoption of Technologies

- Conclusions
SPIRE PPP

Sustainable Process Industry through Resource and Energy Efficiency

Comprises 8 sectors

First-ever Public Private Partnership (PPP) for process industries

R&I projects planned for ca. 2 billion € (over 7 years)

> 50 currently running projects
144 industry and research members
Why a PPP and why SPIRE?

- **Prioritisation** of R&I in line with the *Europe2020* objectives and industry needs

- **Solve** problems **together**:
  - Strongly commit industry to **joint objectives**
  - Find **synergies** across sectors
  - Create the basis for **uptake in industry**

→ Research and innovation will **generate a tangible impact** (EU wide) in the **8 industry sectors**

→ **Strengthen** European industry’s **competitiveness**

*Please visit the SPIRE booth for more information!*
SPIRE Vision & Roadmap

Towards Tomorrow’s Scenario:

- (Re)invent feedstock (waste, bio, CO₂)
- Reduce emissions; (re)invent energy & resource management concepts, incl. industrial symbiosis
- Introduce digital devices for better monitoring and control
- (Re)invent materials for optimised processes
- (Re)invent processes & materials with a significantly increased impact on resource & energy efficiency down the value chain: transport, housing
- Reduce waste & (re)invent technologies for valorisation of waste streams within and across sectors
Call “Integrated Process Control”
(SPIRE-2014-01)

Data treatment & mining:
- Decision support
- Process optimisation
- Integration methodologies
- Soft-sensors

Sensors:
- Fast inline
- Disposable
- Miniaturized
- Swarm sensors
- Spatially resolved

Closed-loop control:
- For intensified / flexible processes
- For disposable sensors
- Models for improved process control

Processes
- Intensified
  - Modular
- Continuous
- Flexible
  - Batch

➔ Increased resource and energy efficiency
➔ Less greenhouse gas emissions
➔ Increased competitiveness
➔ High quality standards
## 5 Projects on “Integrated Process Control”

<table>
<thead>
<tr>
<th>Projects</th>
<th>Beneficiaries</th>
<th>EU-Funding</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Integrated Control and Sensing for Sustainable Operation of Flexible Intensified Processes</strong></td>
<td>15 (7 process industry)</td>
<td>6.0 million €</td>
</tr>
<tr>
<td><strong>Integrated Process Control based on Distributed In-Situ Sensors into Raw Material and Energy Feedstock</strong></td>
<td>15 (5)</td>
<td>6.0 million €</td>
</tr>
<tr>
<td><strong>In-line Cascade Laser Spectrometer for Process Control</strong></td>
<td>9 (1)</td>
<td>5.6 million €</td>
</tr>
<tr>
<td><strong>Robust and affordable process control technologies for improving standards and optimising industrial operations</strong></td>
<td>17 (4)</td>
<td>5.5 million €</td>
</tr>
<tr>
<td><strong>Cross-sectorial real-time sensing, advanced control and optimisation of batch processes saving energy and raw materials</strong></td>
<td>10 (3)</td>
<td>6.0 million €</td>
</tr>
<tr>
<td></td>
<td>66 (20)</td>
<td>29.1 million €</td>
</tr>
</tbody>
</table>
The Concept of CONSENS

**Mission:** Advance the production of high-value products that meet high quality demands in flexible intensified continuous plants.

**Characteristics**
- Miniaturized equipment
- Intensified heat & mass transfer
- Possibly modular setup

**Benefits**
- Product uniformity
- Sustainability
- Fast adaption to market demand
- Innovative products

**General needs:**
- Fast inline & miniaturized sensors
- Closed-loop control of flexible intensified continuous processes
- Online monitoring & engineering of PAT-based control
MAKING THE PROCESS ANALYTICAL TECHNOLOGY (PAT) INITIATIVE A REALITY FOR THE EUROPEAN INDUSTRIES THROUGH INTEGRATED PROCESS CONTROL

PAT Benefits:

- Greater production capacity
- Easier and more predictable processing
- Less waste generated
- Cost savings

Industry challenges:

- High costs of analysers
- Lack of a flexible platform for the integration of different analysers into existing plants
- PAT generates large volumes of data that need to be managed
The Concept of ProPAT

- Integrated process control
- low cost smart sensors and real time monitoring
- Application in process industries.
  - Minerals, Ceramics, Non-ferrous metals, and chemical
PROPAT GRANULOMETER (PROG)
ONLINE PARTICLE ANALYSER

- One order of magnitude lower price than competitors
- Millions of particles measured per shoot. Large Field of View (FOV) + high speed particle flow allow more representative statistics.
- Particle size range customized by design
- Sensor in proximity to the sample allows measurements of high concentrated particle flows.
- Suitable for in-line or at-line operation

ProG is an affordable and easy integrable particle size analyser for online PSD monitoring of industrial powders. It measures other optical parameters for online QC of solid and liquid samples:

- Particle Size Distribution (PSD)
- Transmission
- Haze
  - Size range from 1 to 200 microns [0.5 um/res]
- Clarity
  - Transmission, Haze and Clarity with 0.1% precision
- Turbidity
  - Turbidity sensitivity 5ng/ml.
Advantages over traditional devices

Traditional Size Analyzers

- Several sources and sensors
- Expensive and large device
- Small volume measurement
- Off-line operation

ProG Granulometer

- Only 1 LED, only 1 CMOS sensor
- Affordable and compact (5 times smaller)
- Large volume measurement.
- Suitable for on-line operation
Cost-effective NIR Spectral Sensor

Through Spectral Engines’ patented MEMS (Micro-Electro-Mechanical Systems) solution can be shrink a lab device all the way to miniaturized IoT smart sensors, through material identification based on infrared fingerprints (spectroscopy).

- Low cost
- Compact
- Real-time
- High performance
- Smart and connected
Detector
Tuneable filter

FTIR

Traditional grating

Spectral Engines Fabry-Perot Filter

Size and Cost

100 cm

10 cm

1 cm

Decades

Years

Today

Technological Age

Fixed mirror

Compensator
Beam splitter

Movable mirror

Grating

Detector

Tuneable filter

Detector

Movable mirror

FTIR

Traditional grating

Spectral Engines Fabry-Perot Filter

100 cm

10 cm

1 cm

Decades

Years

Today

Technological Age

Movable mirror

FTIR

Traditional grating

Spectral Engines Fabry-Perot Filter

100 cm

10 cm

1 cm

Decades

Years

Today

Technological Age
About Megara

- Rosin based and other synthetic resins
- Industrial and architectural coatings
- Paint, adhesive, paper and construction industry.

1961

- 50 years in innovative technologies meeting ‘customers' most demanding applications.
- Continued investment in R&D, technical support and new product development.
- Regarded as a most innovative Greek supplier
Products & Markets

- MEGARA: production of saturated polyesters for powder coatings
  - Produce 10000 ton/year, with sales € 24 Million
  - Batch to batch variability: 6-8%
  - Cycle time: 24-30 hours

- ProPAT Aims:
  - Replace manual sample measurements with in-process sensors
    - NIR
    - Viscosity
  - Automated feedback control.
Process Experiments

- 5 different batch runs (2 litre vessel)
  - Multistep reaction
  - Temperature range: 200-240°C
  - Reaction time: 24-30 hours

- NIR spectra and temperature recorded
  - NIR Sensor 1.7 and NIR Sensor 2.2
    5mm pathlength

- Reaction Progress: Offline tests
  - Critical Quality Parameters:
    Hydroxyl, acid value and viscosity
Challenges

Initial mixing and heating up

1st stage  2nd stage

Solids

Bubbles

Control
Raw Data

1\textsuperscript{st} reaction stage

2\textsuperscript{nd} reaction stage

Relative time in 3D graph

Absorbance (AU)

Signal Strength

Raw Data

Absorbance (AU)

Signal Strength

Relative time in 3D graph

Wavelength (nm)

Relative time (min)
Polymerization process – Data analysis

Preprocessing of process spectral data data

- Outlier removal
- Noise filtering
- Baseline correction

Raw NIR data

Preprocessed NIR data

Process time

1300 1400 1500 1600 1700
Wavelength [nm]

0 0.5 1 1.5 2 2.5 3
NIR signal [a.u.]

1300 1400 1500 1600 1700
Wavelength [nm]

-2 -1 0 1 2 3 4 5 6
NIR signal [a.u.]
Polymerization process – Data analysis

On-line prediction of process quality parameters with NIR and multivariate calibration.

NIR information and/or parameter prediction will be used for process control.
- instantaneous measurements of key quality parameters
  - Continuous on-line viscosity and acid/hydroxyl number measurement
  - Systems proven & reliable
  - Operator knows the state of the reaction and the product characteristics at anytime during the batch process

Impact

- Safer procedure
- Improvement of productivity and reduction of operating costs
  - Reduce batch time
  - Greatly reduce the manpower needed
  - Eliminate the manufacturing of off-spec batches and obtain consistent, on-spec product
- Improvement of product quality & new products
  - manufacturing resins with very narrow specification ranges
Impact

- **Economic**
  - 20% reduction of cycle time
  - minimization of the off-spec batches which currently account for 5% of the total annual production

- **Environmental**
  - fossil energy use reduction up to 30% (via optimum process conditions and cycle time)
  - reduction in non-renewable, primary raw material use of up to 20% (by reduction of scrap)
  - CO2 footprint reduction up to 40%
Disruptive business possibilities for NIR

Improvement and automation of industrial production (Industry 4.0)

Improvement of food production (Internet of farming)

Smart devices (Smart homes)

Counterfeits and safety (Field inspection)
Integrated Control and Sensing for Sustainable Operation of Flexible Intensified Processes

EuroPACT – 2017-05-11

Manuel Pereira Remelhe (Bayer AG)

This project has received funding from the European Union’s Horizon 2020 research and innovation programme under grant agreement N° 636942
The Partners in the Consortium

Process industry
- Arkema
- Bayer
- Clariant
- Coatex
- Solvay

Research institutes & academia
- BASF
- Invite
- TNO
- TU Dortmund
- Università degli Studi di Cagliari

Supplier for instrumentation & SMEs
- BAM
- KROHNE
- atlan-tec
- Consens

EuroPACT 2017 – SPIRE Projects with high Impact – Manuel Pereira Remelhe
Results from F³ Factory Project*

» Flexible intensified continuous processes

» Several pilot plants were built

» Average improvements in the Pharmaceuticals and Fine Chemicals Sector:
  • Yield improved by 10%
  • Use of solvents reduced by 2/3
  • Manufacturing costs reduced by 16%

» Technology gap: PAT-based integrated process control solutions that take the specific challenges into account

*Funding from European Commission (FP7/2007-2013) under grant agreement n° 228867
Challenges and Requirements

- Small dimensions → fast and compact sensors
  → fouling and clogging of plant equipment

- Higher quality levels → more accurate sensors

- Complex phenomena → model-based control, online monitoring

- Variety of products, innovative products, & short product life cycles → flexible control methods
  → fast design of control solutions
## CONSENS Case Studies & Novel Technologies

<table>
<thead>
<tr>
<th>Goals</th>
<th>Intensified synthesis of organic compounds</th>
<th>Intensified production of high-viscous polymers</th>
<th>Continuous formulation of complex liquids</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>High product <strong>purity</strong></td>
<td>Tight control of product <strong>viscosity</strong> → less waste, solvent-free process</td>
<td>Tight control of product <strong>rheology</strong> → less waste, water-free product</td>
</tr>
<tr>
<td></td>
<td>Reduce solvent use</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td><strong>Reduce fouling</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Fast transients for start-up, grade changes and shut down</strong></td>
<td>less waste</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Novel sensors</td>
<td><strong>online NMR sensor</strong></td>
<td><strong>inline rheology sensor for polymer melts</strong></td>
<td><strong>inline rheology sensor for complex fluids</strong></td>
</tr>
<tr>
<td>Novel control</td>
<td>Self-adapting &amp; self-optimizing control</td>
<td></td>
<td><strong>Data-driven rheology control</strong></td>
</tr>
<tr>
<td>Tools</td>
<td>Sensor failure detection, performance monitoring</td>
<td></td>
<td>design evaluation</td>
</tr>
</tbody>
</table>
Modular plant for intensified continuous synthesis of a pharmaceutical precursor

Developed in the F³ Factory project*

Container includes:

- Receiver tanks
- Two reaction steps
- Mixer-settler-extraction
- Destillation
- Automation
- Heavy current equipment
- Cooling / heating unit

In CONSENS only the first reaction step is considered

*Funding from European Commission (FP7/2007-2013) under grant agreement n° 228867
Lithiation Reaction

Aniline + 1-Fluoro-2-nitrobenzene (FNB) + Li-HMDS → 2-nitrodiphenylamine + 2HMDS + LiF(s)

» Widely used in the production of pharmaceuticals and fine-chemicals

» Very fast & exothermic reaction

» Typical industrial setup: Batch reaction at -70°C (low energy efficiency)

» Continuous intensified process enables reaction at much higher temperatures with less solvent

Complex mechanisms:

- Aniline + Urea → Urea + Anilino
- Phenyl + Carboxylic acid → Carbamoyl + Phenyl
- Diphenyl + Urea → Diphenylamino + Urea
Specific Challenges of the Case Study

- Miniaturized flexible equipment
- Fast and complex exothermic reaction
- Hazardous and inflammable substances → Explosion protection!
- Prone to fouling & clogging
- Multi-objective constrained optimization

Standardized plant modules (for “Plug & Produce) with instrumentation and local control \( (LC) \)
Why Control is Important

Disturbances:  • Compositions of raw-materials (variations)  
• Effects of fouling (drift and abrupt changes)

Constraints:  • $T_i \leq 60^\circ C$  (shutdown threshold to prevent boiling of solvent)  
• $P_i \leq 20$ bar  (shutdown threshold to prevent mechanical damages)  
• $C_{residues} \leq 0,5\%$  (purity)  
• $F_i \leq 20$ l/h  (maximum pump rates)

Maximize:  • Production rate (kg/h)  
• Concentration of desired product  
• Run time of reactor without cleaning (due to fouling)

Minimize:  • Residues of Aniline and FNB  
• Other impurities (unwanted reaction products)  
• Excess of feed substance LiHMDS  
• Amount of solvent (THF)  
• Cooling power

Prevent:  • Complete clogging of reactor
Highlights: Novel Sensor Technologies

**Inline Fouling Sensor**

- Real-time monitoring of fouling layer thickness and growth
  - Ultrasonic non-invasive sensor
  - Possibility of spatial fouling characterization
  - Bended pipes possible

- Applicable to pipes of reactors, heat exchangers etc.

**Online NMR Sensor**

- Composition measurement by Nuclear Magnetic Resonance spectroscopy
  - Calibration-free operation possible
  - Non-invasive
  - High linearity signal/composition
  - Turbid media
  - Small changes in molecules detectable

- Explosion proof and fully automated setup
Highlights: Novel Monitoring Technologies

Sensor Failure Detection
- Detects faulty sensor data online
- Provides reliable substitute values for faulty sensors
- Based on artificial neural networks
- Short time for model setup

Performance Monitoring
- Automated performance monitoring and data analysis workflow
- Consideration of long term deviations due to fouling, catalyst deactivation or seasonal effects
- Validation if the autonomous control system reaches the optimum
Conceptual Integration of Technologies

Control | Data-usage | Sensors

Model-based control → Online state & parameter estimation → Performance monitoring

FNB → Dos. → Mixing, cooling → Reactor
Aniline → Dos. → Mixing, cooling
LiHMDS → Dos. → Mixing, cooling

Performance monitoring → Sensor failure detection & correction

Other sensors
Fouling Sensor
Online NMR

Model-based Evaluation of PAT-based Control + Process in Design Stage
Technical Integration of Technologies

- DCS
- Performance Monitoring
- Sensor Failure Detection
- Plant-wide optimizing control

Communication: WLAN, Ethernet, OPC-UA, HART

Topic covered in other projects: hardware and software solutions for fast integration of control (sub-)systems of modular plants for “Plug & Produce”
Expected Impact for Manufacturing of Pharmaceuticals and Fine Chemicals

» **Technical:**
  - Enabling the migration of batch to flexible intensified continuous processes
  - Making processes resilient to variances in feed-stocks and external disturbances
  - Enhancing fast process development for new products.

» **Relative savings** (from F³ Factory project):
  - Yield improves by 10%
  - Use of solvents reduces by 2/3
  - Manufacturing costs reduces by 16%

» **Scenario:** 2% of market migrates from batch to intensified continuous plants
  - 130 M€/year cost savings
  - 176,000 t/year less use of solvents
  - 170,000 t/year less CO₂ emissions
Overall Expected Impact of CONSENS

- **Economic:**
  - > 130 M€/year in pharma & specialty industry
  - > 100 M€/year in consumer chemicals
  - > 35 M€/year for specific polymers

- **Environmental:**
  - Reduction of CO₂ emissions
    - 230,000 t/year for specific polymers
    - 170,000 t/year in pharmaceutical & specialty industry
  - 176,000 t/year less use of solvents
Adoption of Novel Technologies

Technologies are not limited to intensified continuous processes:

- Novel sensors are applicable to all kind of “fluid processes” where the specific capabilities are needed (batch or continuous).
- Control and monitoring methods, as well as design evaluation are applicable to all kind of continuous processes.

Identification of further opportunities already started

1. Industrial partners of CONSENS
2. Chemical industry as a whole
3. Cross-sectorial (e.g. food & beverages, cement, waste water, engineering, ...)

As soon commercial versions are available, process industry will adopt novel technologies.
Sensors

- Inline rheology sensor and online NMR have very promising market prospects.
- Fouling sensor developed by TNO will probably be used for special applications.
- Krohne Messtechnik GmbH is a supplier of instrumentation and will follow up.
- Providers of lab-NMR-equipment want to address needs of process industry.
- Patents and NMR-software-packages will be licensed.

Tools and services:

- Sensor failure detection: atlan-tec Systems GmbH achieved already a high TRL and will integrate the technology into their APC suite.
- Control solutions: Results are being published so that everyone can use it.
- Performance monitoring: will be used at Clariant and is being published
- Design evaluation: will be used at Bayer and will be published
Conclusions

- **ProPAT and Consense:**
  - Novel sensing and control technology and Integrated Process Control has a huge potential impact; beyond spire
  - strong participation of industrial partners promising significant industrial uptake
  - A targeted approach to commercial exploitation.

- **SPIRE PPP**
  - Drives Process R&I for Societal change
  - Enhances and promotes Processing industry’s impact on Europe
  - Increased competitiveness and reduced environmental foot print
  - sustainability of European process industry.
Thank you!


Frans Muller  Manuel Pereira Remelhe
F.L.Muller@Leeds.ac.uk  Bayer AG
Leeds University  Germany
UK

Disclaimer: This document is provided with no warranties whatsoever, including any warranty of merchantability, non-infringement, fitness for any particular purpose, or any other warranty with respect to any information, result, proposal, specification or sample contained or referred to herein. Any liability, including liability for infringement of any proprietary rights, regarding the use of this document or any information contained herein is disclaimed. No license, express or implied, by estoppel or otherwise, to any intellectual property rights is granted by or in connection with this document. This document is subject to change without notice.

This document reflects only the view of the author(s) and the European Commission cannot be held responsible for any use which may be made of the information contained herein.