RHODOLIVE Kick-off meeting

Presentation of partners: National Institute of Chemistry
dr. Miha Grilc
more than 70 years of tradition
National institute of Chemistry in collaboration with stakeholders

- Expanding knowledge of chemistry and associated studies
- Transferring knowledge to younger generations
- Applying newly acquired knowledge to industry

- 25 EU projects
  - 13 H2020
  - 4 Interreg
  - 1 CRP ICGBE
  - 2 NATO
  - 4 ERA NET
  - 50 ARRS
  - 1 EDA

- 25 EU projects

Rhodolive kick-off meeting | Burhaniye | 5.6.2018
Department of Catalysis and Chemical Reaction Engineering

Research topics

- Research subfield: Carbon dioxide activation
- Research subfield: Methane activation & conversion
- Research subfield: Pharmaceutical process engineering
- Research subfield: Biomass-derived building blocks

Ongoing projects

Horizon 2020:

- MefCO2, Synthesis of Methanol from Captured Carbon Dioxide Using Surplus Electricity (SPIRE-02-2014)
- Fresme, Methanol from CO2 Blast Furnace gasses to be used as ship transportation fuel
- nextBioPharmDSP, Next-generation Biopharmaceutical Downstream Process (BIOTEC-4-2014)

ERA-NET:

- Mar3Bio, Biorefinery and Biotechnological Exploitation of Marine Biomasses (MarineBiotech - Marine Biotechnology ERA-NET)
- RHODOLIVE, Biovalorization Of Olive Mill Wastewater To Microbial Lipids And Other Products

NATO SPS:

- 984738, Enhanced Portable Energetically Self-sustained Devices for Military Purposes (ESCD - Emerging Security Challenges Division); coordinator

COST Actions: 3

Members of EERA – European Energy Research Alliance

Slovenian Research Agency projects: 1
Bilateral cooperation projects: 2

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Methanol fuel from CO₂

Horizon 2020 (MefCO₂, 2014-2018, 11 mil. EUR in total)

MefCO₂ aims to produce green methanol as energy vector from captured CO₂ and hydrogen produced using surplus renewable energy. Goal 2018: 1 ton / day
Research subfield: **Carbon dioxide activation**

–Computational fluid dynamics

- generation of packed bed reactors’ random and realistic geometry model
- generation of models for various packing sizes and shapes
- CFD simulations of flow inside generated models for various Reynolds numbers
- kinetic Monte Carlo simulations of adsorption, desorption and surface chemical reactions
From Residual Steel Gases (CO$_2$) to Methanol

Horizon 2020 (*FReSMe, 2016-2020*, 11.4 mil. EUR in total)

- Lab developments supporting the technology improvement
- Modelling and simulation in order to optimize the processes
- Pilot plant construction and test campaigns implementation
- Life cycle analysis to evaluate the actual reduction achieves on GHG emissions
- End user demonstration using the methanol produced in a Ferry
Adaptable Reactors for Methane Valorisation

Horizon 2020 (ADREM, 2015-2019, 6 mil. EUR in total)

Development of plasma-activated (catalytic) systems for enhanced methane coupling processes

- Catalyst preparation, characterization and testing
- Process control and instrumentation
- Reactor development
- TRL 5 setups, construction, validation and roadmapping
- Concept evaluation
  - initial reactor/chemistry benchmarking
- Concept evaluation
  - process performance benchmarking
- Concept evaluation
  - techno-economic assessment
- Concept evaluation
  - feasibility, adaptability and safety assessment

Gas flow in
Gas flow out/products

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Next-generation biopharmaceutical downstream process

Horizon 2020 (nextBioPharm LEIT, 2015-2019, 11 mil. EUR in total)
- Chromatography modelling (adsorption/desorption, mass transfer, thermodynamics)
Biomass-derived building blocks subgroup

- 8 Postdocs, 5 PhD students

Ongoing activities

**LIGNOCELLULOSIC BIOMASS:**

- Fractionation
- Liquefaction
- Furfural synthesis from hemicellulose
- Adipic acid synthesis from glucose
- Isolation of flavonoids from fir bark
- Levulinic acid valorisation by HDO
- Lignin valorisation by HDO

**MARINE BIOMASS:**

Biorefinery and Biotechnological Exploitation of Marine Biomasses (MarineBiotech - Marine Biotech. ERA-NET)

- Extraction of chitin from crustaceans
- Chitin conversion into chitosan and chitlac
- Isolation of oligosacharides from algae
Research subfield: **Biomass-derived building blocks**  
– Extraction of valuable bio-chemicals

- **BIO COUP Project**: Co-processing of upgraded Bio-Liquids in Standard Refinery Units (17 partners, 8 countries)
- **Isolation and fractionation** technology for the production of discrete target oxygenated compounds or defined fractions
- Utilization of these compounds to **derive industrial chemicals and products**, evaluated by companies (CHIMAR Hellas, Greece)

2. EU project (BIOCOUP-Contract no 518312 (SES6), 2006, 5 years, 7.7 MEur)
Research subfield: **Biomass-derived building blocks**

– 1\textsuperscript{st} generation lipid biofuels

- transesterification of vegetable oils
- novel kinetic model based on different glyceride and fatty acid ester composition
- integration of thermodynamics, fluid mechanics, transport phenomena and chemical kinetics
- batch, continuous and membrane reactor operation using homogeneous, heterogeneous inorganic and enzymatic catalysis

Research subfield: **Biomass-derived building blocks**

- 2\textsuperscript{nd} generation lignocellulosic biofuels
  
  - selective fractionation processes
  - depolimerisation of whole lignocellulosic biomass
  - catalytic conversion of liquefied biomass to fuel
  - kinetic models developed for decomposition and HDO
  - screening of 30 synthetized and commercial HDO catalysts
Research subfield: **Biomass-derived building blocks**

– Discarded potentials of lignocellulosic biomass

Slovenian smart specialization strategy (CELCYCLE, 2016-2020, 8.6 mil. € in total)

5 conceptual sections:

1. **Valorisation of biomass potential and development of bio-based products: nanocellulose and green chemicals**

2. Development of advanced and multifunctional materials with integrated nanocellulose and environmentally acceptable additives: paper, board and yarn

3. Development of products with higher proportion of bio-based components and improved functionalities: construction, automotive, textile and electrical industry

4. **Development of procedures for biological and mechanical processing of solid waste into products with added value**

5. Development of innovative system for energy recovery from waste

**Intersectoral and social networking with emphasis on local production circles – new value chains**
INDUSTRIAL FURFURAL PRODUCTION FROM HEMICELLOUSE

Co-operation with industrial partner

EXTRACTIVES

HEMICELLOUSE

Lignocellulosic Biomass

+H₂O, H⁺

+H₂O

+Solvent, H⁺

PHARMACEUTICALS FROM EXTRACTIVES

LEVULINIC ACID
γ-VALEROLACTONE
PENTANEDIOL (1,2; 1,4; 1,5)
GLUCARIC ACID
ADIPIC ACID
SORBITOL

Aromatic and Cyclohexanoic Compounds

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High-throughput experimenta- \textit{in silico} approach

High-throughput experimental measurements
- Fast experimental screening
- Systematic experimental design
- Online process analysis

Analytics
- Identification
- Quantification
- Processing

Mechanism
- Intermediates
- Pathways
- Reactions

Density functional theory

Model
- Mass transfer (gas-liquid-solid)
- Adsorption/desorption processes on material surface
- Surface reactions based on elementary steps

Characterisation
- Composition
- Structure
- Morphology

Thermodynamics

Kinetic Monte Carlo

- Characterized developed material
- Structure–activity correlation
- Structure–selectivity correlation
- Reaction/process mechanism
- Effect of process conditions
- Micro-scale process model


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Our profile:
- R&D on a lab scale
- Scale-up (based on combined experimental and in-silico studies)
- R&D on a pilot scale
- Unit operations
- Kinetic modeling, parameter estimation and sensitivity analysis
- Process simulation and optimization (CFD, DFT, Monte-Carlo, Aspen+)
- Downstream processing

Our role in a Rhodolive:
- WP 2
- Separation process development (lipids, carotenoids, biophenols)
- Pre-treatment, extraction, isolation, purification, solvent recovery
- Experimental screening of operating conditions
- Modelling of thermodynamics and mass transfer kinetics
- Sensitivity analysis and process optimization
- Purified VCP’s to AIDISA
- Isolated phenolics to UNI Kassel
Our profile:

- R&D on a lab scale
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- Process simulation and optimization (CFD, DFT, Monte-Carlo, Aspen+)
- Downstream processing

AVAILABLE RESEARCH INFRASTRUCTURE:

High-throughput high-pressure reactor systems:
- 5 x parallel continuous fixed-bed reactors (in-house fabricated)
- 6 x parallel fully-automatized stirred reactors (Amar)
- 6 x parallel carousel Plus Reaction Station (Radleys)

Individual high-pressure reactor equipment:
- 1 x Fully automatized fixed bed or trickle bed reactor (PID Eng&Tech)
- 2 x 1700 mL stirred reactors (Parr, SS-316 and Titanium)
- 1 x 300 mL stirred reactor (Autoclave engineers)
- 1 x Reactor equipped with a scale for thermogravimetric analysis (Rubotherm)
- 1 x RC1e calorimeter with in-situ FTIR and FBRM probes (Metler Toledo)
- 1 x Fully automatized view-cell with highly-precise T and P sensors (in-house fabr.)

Analytical equipment:
- 4 x Two-module μ-GC (2 x Agilent, 2 x Inficon)
- 4 x GC–TCD (2 x SRI, 2 x Agilent)
- 2 x GC–MS/FID (1 x Agilent, 1 x Shimadzu)
- 1 x GC–FID (Thermo Scientific)
- 2 x HPLC/DAD (HP/Agilent, Thermo Scientific)
- 1 x UHPLC/DAD–RI (model known in May)
- 2 x FTIR with DRIFT, ATR liquid–probe & gas–cell modules
- 1 x UV-VIS spectrophotometer (Perkin–Elmer)
- 1 x Benchtop NMR (Magritec)

Glass apparatus:
- 1 x Labmax (Metler Toledo)
- 1 x Ebuliometer (Fischer Sci)
- 2 x Pilot scale adsorption and distilation units

Characterization equipment:
- 1 x Physisorption analyzer (Micromeritics ASAP 2020)
- 1 x Chemisorption analyzer with MS (Micromeritics AutoChem)
- 1 x Co-owners of scanning electron microscope (Carl Zeiss)
High-throughput reactor systems

6 × parallel carousel Plus Reaction Station
- Radleys
- 100 ml
- Tmax = 180 °C

5 x parallel high-throughput packed bed reactor
- In-house built
- 5 mL, SS-316
- Max P = 200 bar
- Max T = 500 °C

6 x parallel stirred autoclave system
- Amar Equipments
- 250 ml
- Max T = 350 °C
- Max P = 200 bar
- SCADA software (fully automatized)
Individual reactor systems

- Batch, Semi-batch, CSTR mode
- G-L, G-S, L-S, G-L-S reactions
- Reactions, (supercritical) extraction
- Online GC and FTIR analysis

Autoclave engineers, 300 mL, SS-316, Max $P = 345$ bar, Max $T = 510 \, ^\circ C$

Parr instruments, 1900 mL, Ti & SS-316, Max $P = 200$ bar, Max $T = 400 \, ^\circ C$

High pressure phase-equilibrium cell (In-house made)
RC1e reactor/calorimeter with *in-situ* FTIR and FBRM (Mettler Toledo)

3L and 200 ml

In situ FTIR and FBRM probes
Analytical equipment

- UHPLC–DAD/RI and fraction collector (Thermo Fisher Scientific)
- 2x HPLC–DAD (Agilent; Thermo Fisher Scientific)
- 2x FTIR/DRIFT/ATR (gaseous/liquid/solid) (PerkinElmer)
- UV–Vis (liquid/solid) (PerkinElmer)
- 2x GC–MS/FID (1 × Agilent, 1 × Shimadzu)
- 4x GC–TCD (2 × Agilent; 2 × SRI Instruments)
- Micro GC (2 × Agilent; 1 × Inficon)
- TOC
- High pressure TG-GC-MS
- Benchtop NMR (Magritek)
- FBRM (Metler Toledo)
Thank you for your attention!

Let's kick off summer with a holiday weekend that isn't warm enough for summer activities.

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