# From sludge drying follow-up using X-ray microtomography to LCA of processes: 13 years research at the LGC

LABORATORY of CHEMICAL ENGINEERING

Processes and Sustainable development

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Introduction to the Laboratory of Chemical Engineering





## University of Liège, Belgium

9 faculties, 1 institute, 1 school





Арр





Law and Criminology school



**Veterinary Medecine** 



Sciences



Med





Management School - University of Liege

**Psychology and Education** 



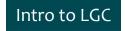
Architecture

✓ 194 masters✓ 68 complementary masters

#### Human and Social Sciences







✓ 38 bachelors

## **Faculty of Applied Sciences**

- 4 departments
  - Aerospace and Mechanical Engineering
  - ArGEnCO = Architectural, Geological, Environmental and Civil Engineering
  - Electrical Engineering and Computer Science

Applied Chemistry





Intro to LGC

## **Department of Applied Chemistry**

- 3 groups About 60 people
- Analysis and Synthesis of Chemical Systems Cryotechnology laboratory
  - (Pr G. Heyen, Pr J.-L. Bozet, Dr M.-N. Dumont)
- Catalytic and electrochemical engineering Nanomaterials

(Pr J.-P. Pirard, Pr B. Heinrichs, Dr N. Job, Dr S. Lambert, Dr C. Gommes)

Chemical Engineering

(Pr M. Crine, Pr D. Toye, Pr A. Léonard)

http://www.chimapp.ulg.ac.be/







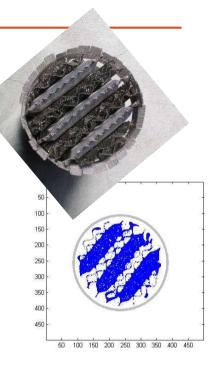
## LGC Research topics

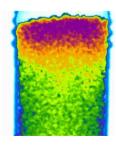
Hydrodynamics in polyphasic systems
 o Packed columns and trickled bed reactors
 b absorption, distillation, reactive distillation, ...
 o Bubble columns

♥ water treatment, G/L contactors, ...

- Mixing in stirred (bio)reactors
   o Study of rheologically complex fluids
- Treatment of gas streams

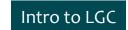
   o VOC adsorption on activated carbons
   o Aerosols capture
   o Gas cleaning











## LGC Research topics

- ✓ Treatment of waste
  - o Biomethanation
  - o Drying



- ✓ Convective drying of deformable materials
  - o Residual sludges o Resorcinol-formaldehyde xerogels
- $\checkmark$  Characterization of porous materials using  $\mu CT$
- ✓ LCA studies of processes
- ✓ Set up of downstream processes o Settling → dewatering → drying







## In brief ...

2008

...

**1998** Chemical Engineer - University of Liège

**FRS-FNRS Research Fellow:** convective drying – sludge – X-ray µCT

2003 PhD Thesis: Study of wastewater sludge convective drying: texture follow-up using X-ray µCT

2004 FRS-FNRS Postdoctoral Researcher: convective drying – carbon xerogels - modeling

Extension of X-ray µCT applications

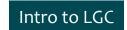
Postdoc research stay in Bordeaux (Laboratoire 'TREFLE')

FRS-FNRS Research Associate: drying – relations between process/product

**2009** Academic position at ULg: Processes and Sustainable development

**Development of existing LCA activities** 







## 3 main research topics

- Drying of deformable materials
  - Both experimental and modeling approaches
  - Long expertise in sludge drying
  - Relation between drying process and product quality
- Characterization of porous materials by X-ray microtomography
  - Initially developed to follow sludge texture during drying
    - Cracks, shrinkage, moisture profiles
  - Extension to different types of cellular materials
  - Now used for product-oriented-engineering approach
- Environmental management: Life Cycle Assessment studies, environmental reporting







About drying ...





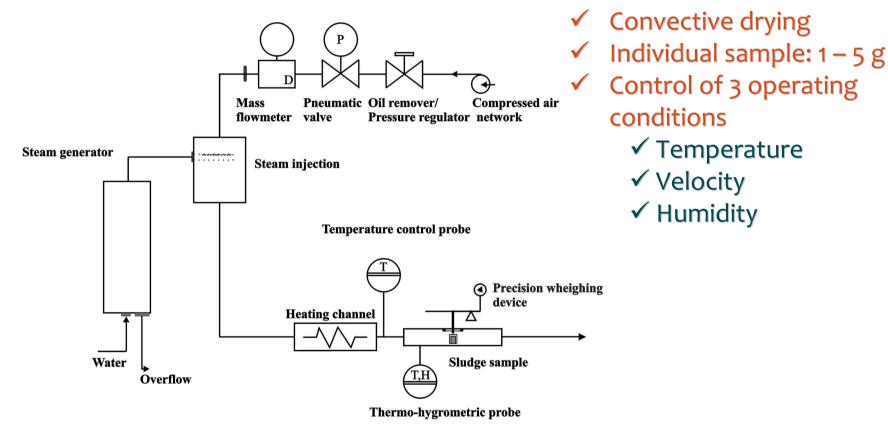
## Convective drying of deformable materials

- Thermal drying = widely used separation process
  - High energy consumption : 10 to 15% of Europe industrial energy use
  - Need for process optimisation
    - Material behaviour
    - Relevant drying models
- Impact of drying on quality of dried product
  - Shrinkage, cracks, moisture profiles, ...
  - Need of characterization tools
- Focus on two main types of materials
  - Sludges
  - Resorcinol-formaldehyde xerogels
- Both experimental data and modeling approach

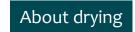




### Micro-dryer

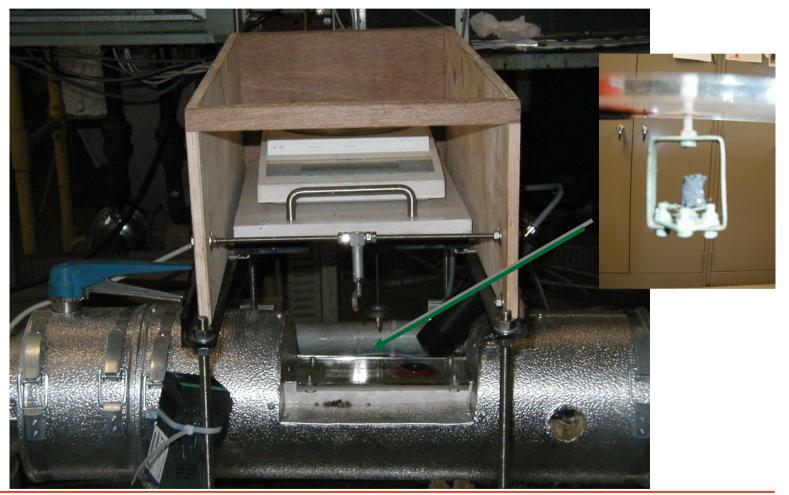








#### 









### Pilot-scale dryer



Fixed bed (cross flow)

- Capacity
- 1 to 3 kg
  Up to 200 kg water/m<sup>2</sup>h

Sevar pilot scale dryer





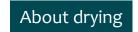


### Veolia dual scale dryer



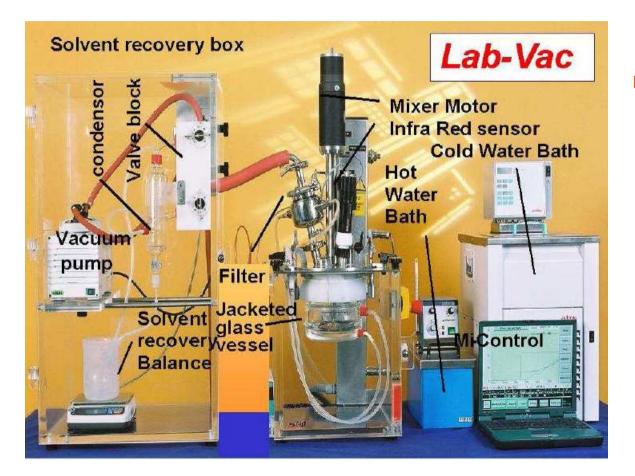
- Small cell
- Fixed bed (cross flow)
- Gas line analyzer







Vacuum agitated contact dryer



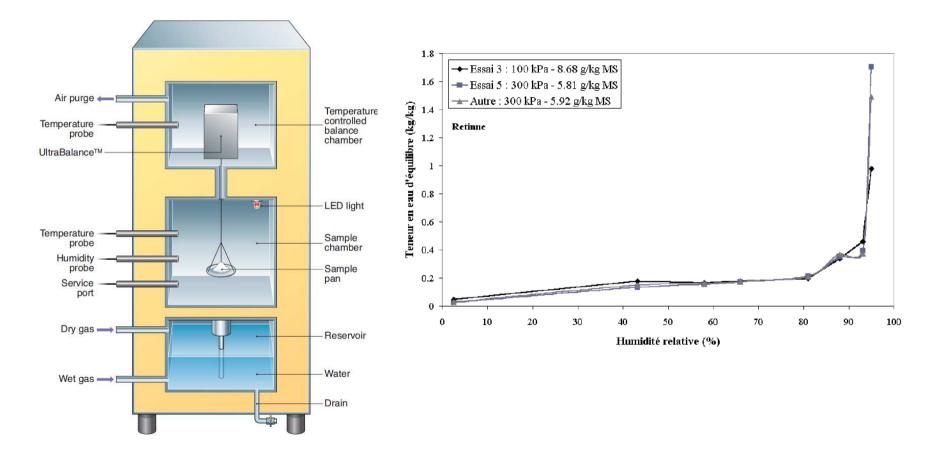
 Thermosensitive products



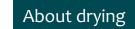




#### Dynamic vapour sorption







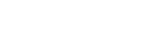


- X-ray tomographs
  - Non destructive 3D imaging technique
  - Follow-up of sample texture
    - External exchange area  $\rightarrow$  drying kinetics
    - Cracks  $\rightarrow$  drying quality
    - Internal moisture profiles  $\rightarrow$  model validation

About drying

■ Sludge bed permeability → sludge rheology







Microtomograph

Purchase year: 2000



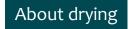
- Source: 40 kV 1 mA Cone beam
- Detector: 768 x 576 pixels

8-bit CCD Camera

- Pixel size: 41 µm
- Max sample size: Ø: 30 mm h: 25 mm







Microtomograph

Purchase year: 2006

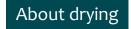


- Source: 100 kV 250 mA Cone beam
- Detector: 4000 x 2300 pixels
  - 12-bit CCD Camera
- Pixel size: from 34 to  $\approx$  2-3  $\mu$ m
- Max sample size: Ø: 35 mm (68 mm with camera offset)

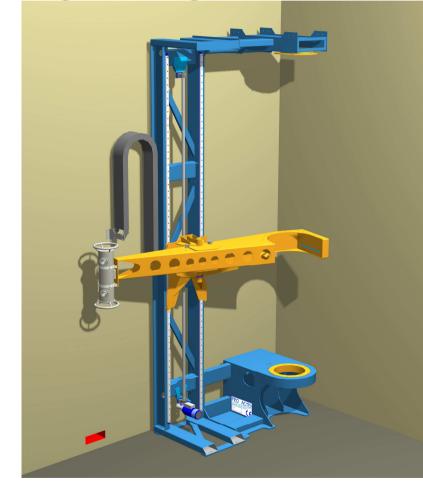
h: 35 mm (70 mm with camera offset)

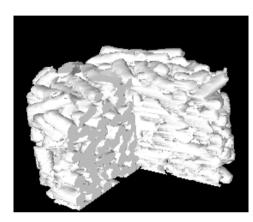


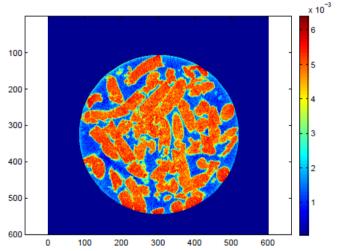




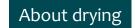
### High energy 'macro' tomograph













## About sludge drying

- Origin of urban residual sludges
  - Activated sludge wastewater treatment plant (WWTP)
  - Production of excess sludge during biological process



Oupeye - 446 500 PE

www.aide.be







## About sludge drying

- Sludge processing within a WWTP
  - Applied to excess biomass produced by the biological treatment
    - Thickening
    - Stabilisation
      - Liming
      - Digestion (biomethanation)
      - Mechanical dewatering
        - Centrifugation
        - Belt filter
        - Press filter

Drying

Valorisation: in agriculture or for energy recovery

Europe : About 50 to 60 million tons wet sludge/year





## About sludge drying

- Sludge drying  $\rightarrow$  several advantages
  - Mass and volume reduction
  - Stabilisation Hygienisation
  - Texture improvement
  - Increase of calorific value
- Sludge drying = complex unit operation
  - Depends on sludge properties
    - Composition, rheology, treatment, storage conditions, …
  - Depends on operating conditions
  - Produces gaseous emissions (pollutants, odors, ...)
  - Highly energy consuming
    - Needs global energy optimization on the process site





Flanders 32% sludge dried in 4 dryers 2 fluidised bed dryers 2 stage dryers



## A lot of investigated topics ...

Drying kinetics Discontinuous pilot scale convectif dryer

Léonard & Crine, IDS 2000 (CD-ROM)

Influence of sludge origin on drying kinetics

Léonard et al., Env Tech, 25, 1051-1058 (2004)

Study of gaseous emissions related to sludge drying

Fraikin et al., ECSM 2010 Fraikin et al., SFGP 2009 Follow up of texture, shrinkage, humidity profiles, at the extrudate scale

Léonard et al., Drying Tech, 20, 1053-1069 (2002) Léonard et al., Drying Tech, 21, 1507-1526 (2003) Léonard et al., Drying Tech, 22, 1695-1708 (2004) Léonard et al., Can J Chem Eng, 83, 127-131 (2005)

Influence of backmixing on drying kinetics (fixed bed)

Léonard et al., IDS 2006, Vol B, 767-772 (2006)

Influence of liming and mixing on drying kinetics

Léonard et al., ECSM 2008, 2010

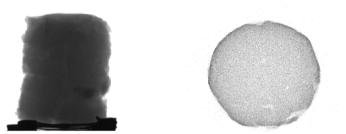


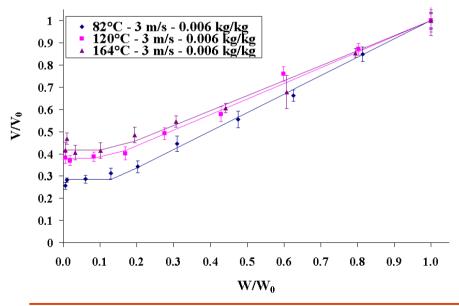


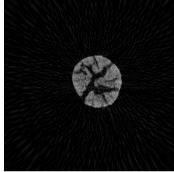
About drying

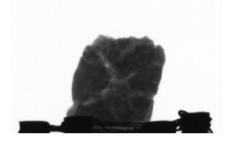
## Follow-up of structural changes

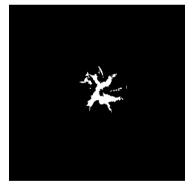
- Shrinkage = necessary to study drying mass flux
- Cracks in relation with quality











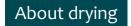
Cracks

Height

X-ray microtomograph Micro dryer scale

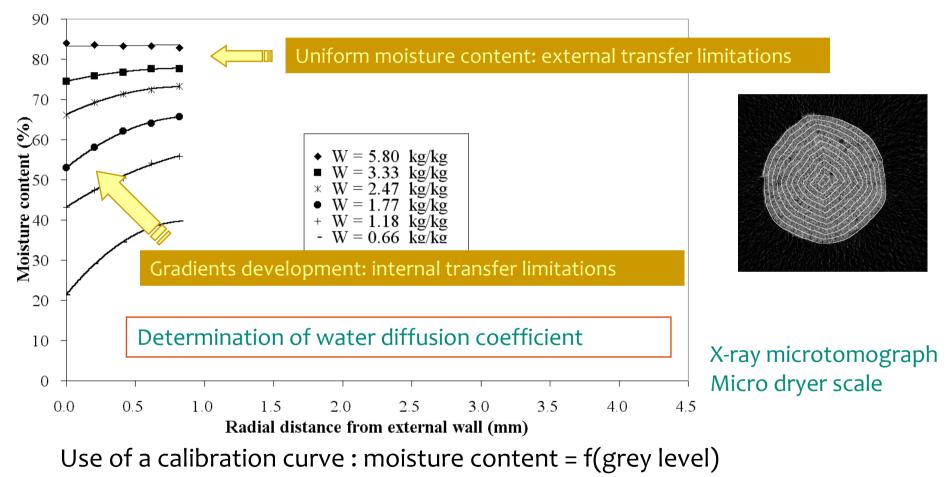






## Follow-up of internal moisture profiles

### Understanding of mass transfer + model validation

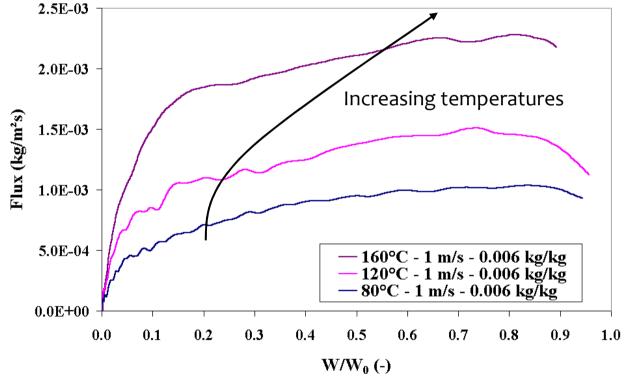






## Impact of operating conditions

### Influence of temperature



Microdryer

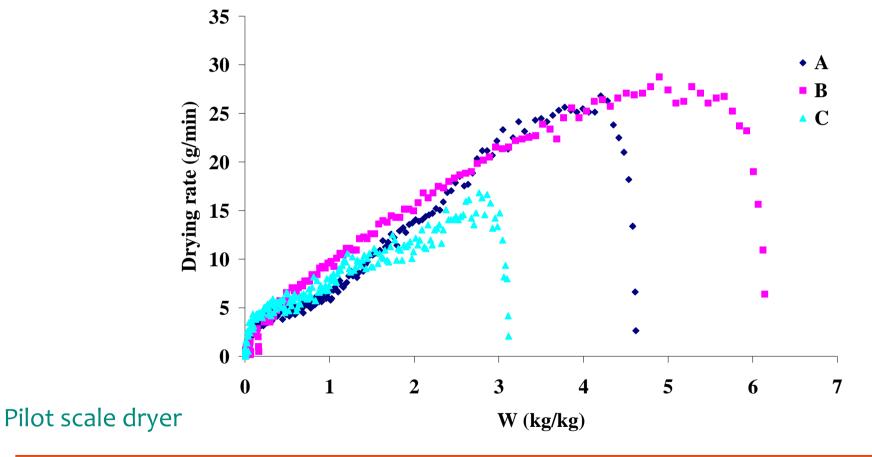






## Impact of sludge origin

Max drying rate: ratio up to 3:1



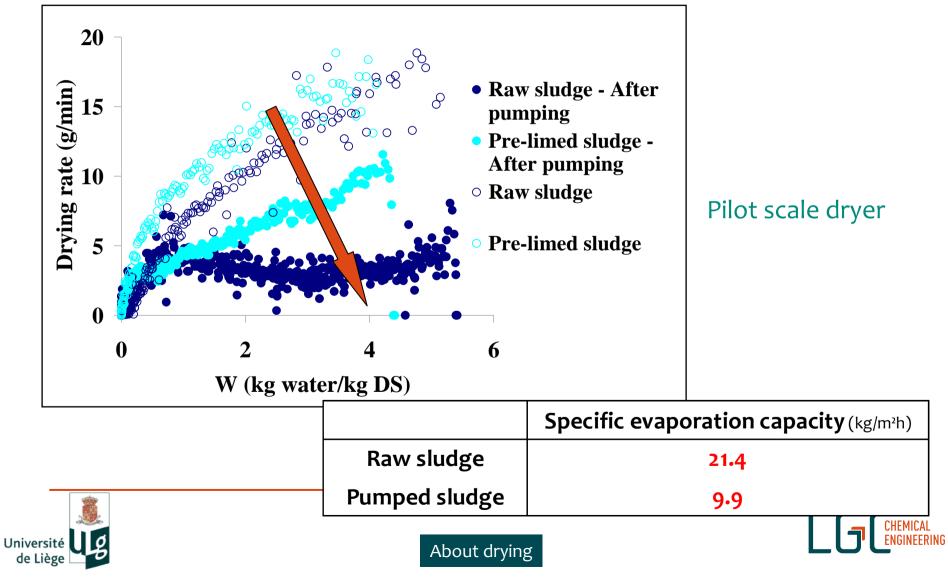


About drying



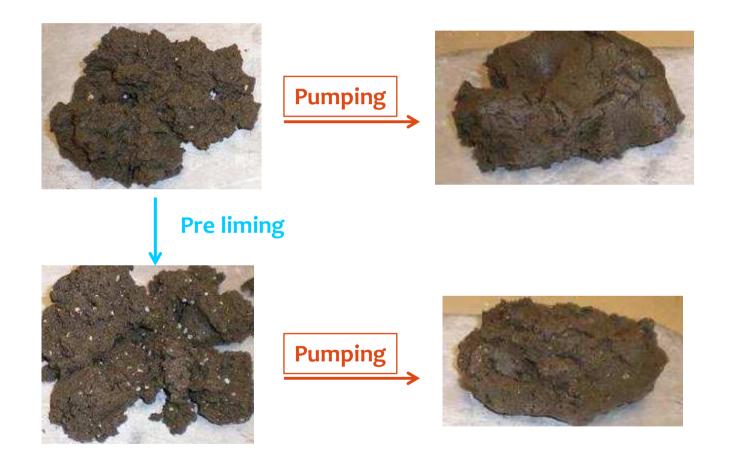
## Impact of sludge pumping

Influence of pumping on drying kinetics



## Impact of sludge pumping

### Importance of textural properties



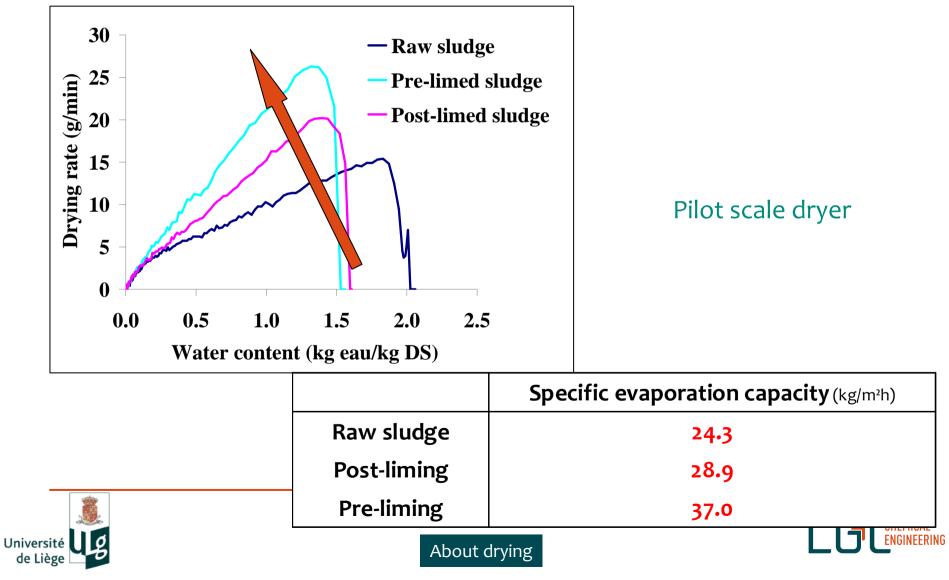






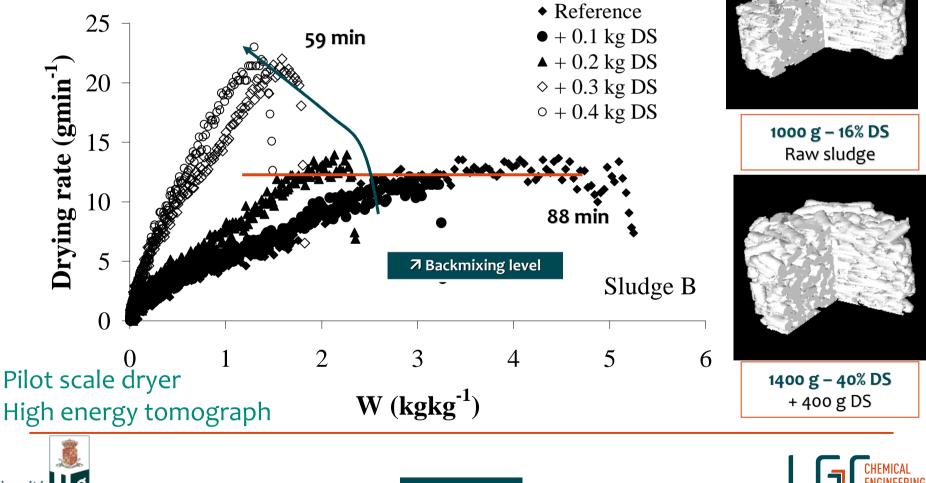
## Impact of sludge liming

Influence of liming on drying kinetics

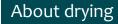


## Impact of back-mixing

■ Recirculation of dried product → way to correct 'bad texture'

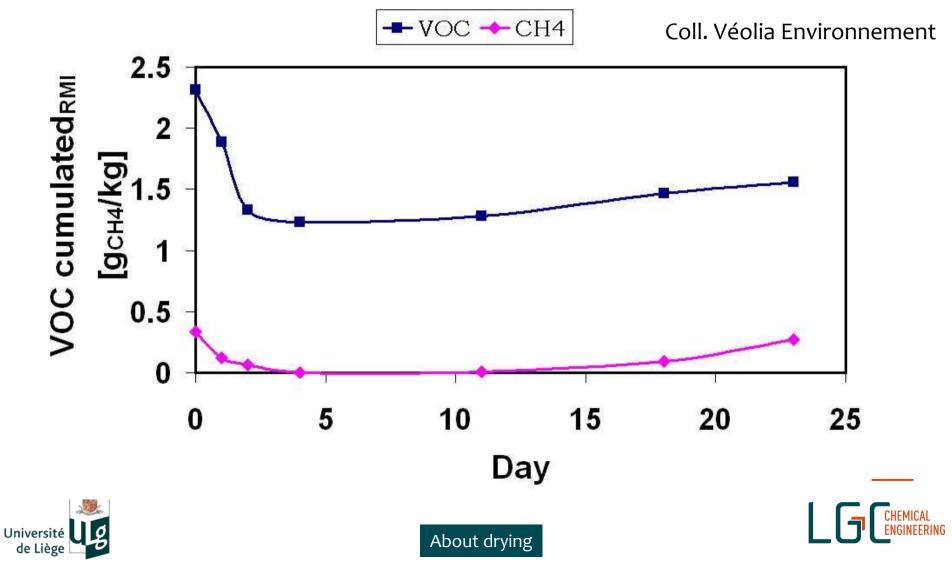






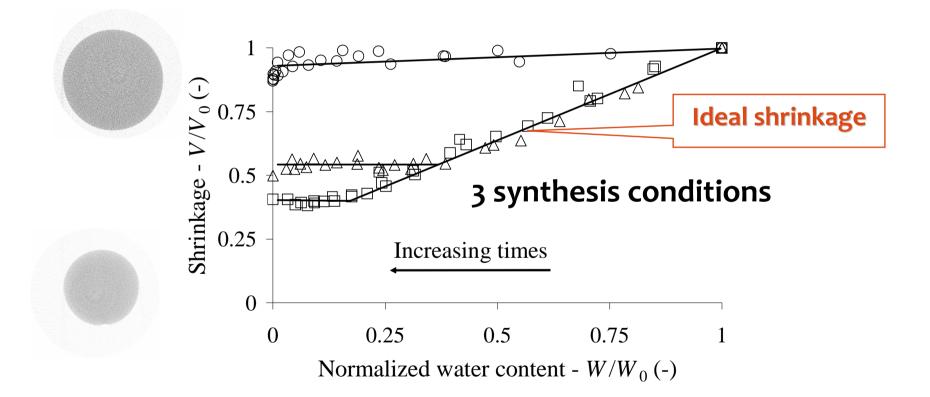
## Follow-up of gaseous emissions

Evolution of VOC with storage duration digested sludge



## About RF xerogels drying

- RF resins = model material
  - degree of shrinkage can be easily controlled
  - tunable pore texture

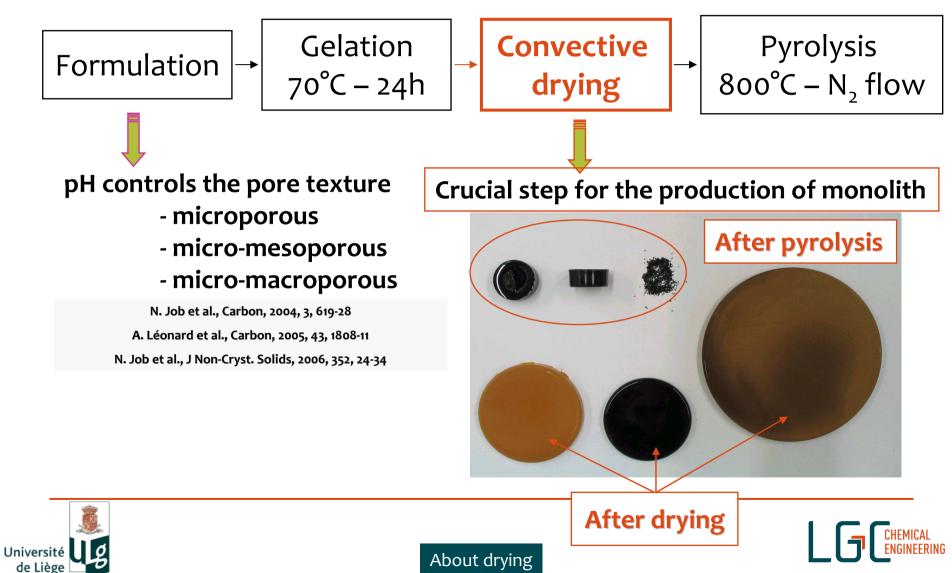






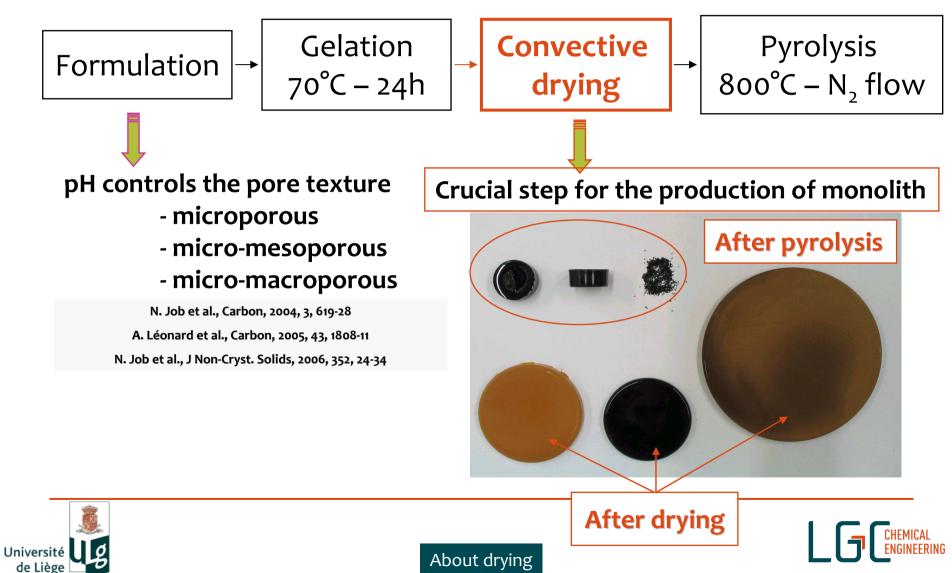
## About RF xerogels drying

RF resins = precursors of carbon materials

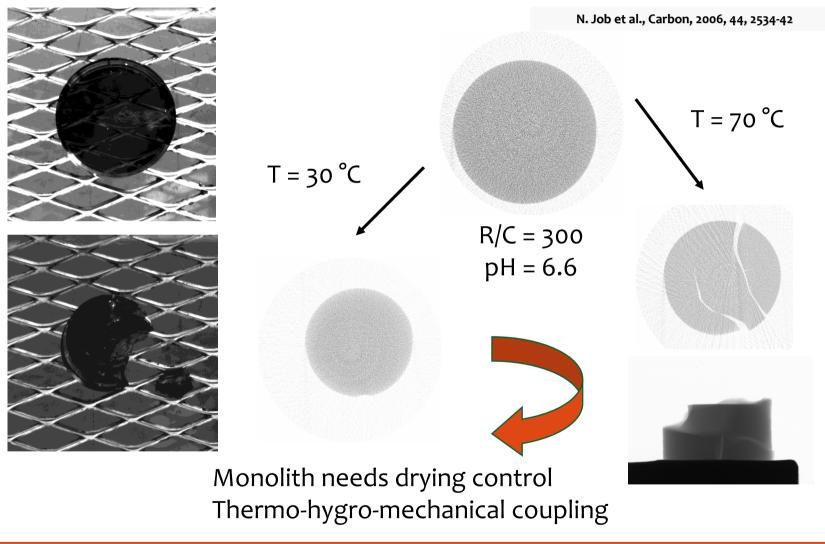


# About RF xerogels drying

RF resins = precursors of carbon materials



### About RF xerogels drying



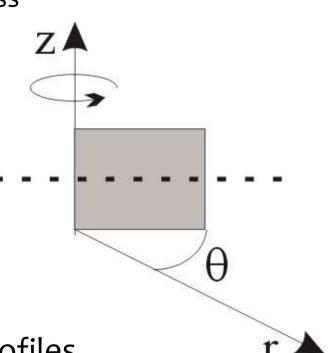






# **Thermo-hygro-mechanical simulation**

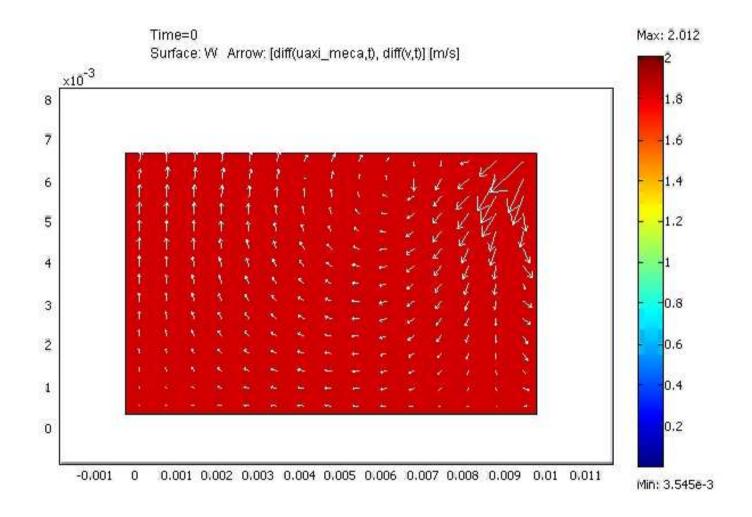
- Development of drying simulation model
  - Gel = ideal shrinking medium
  - Saturation throughout the drying process
  - Eulerian coordinates + solid velocity
    - mechanical coupling
  - ALE method for resolution
    - = Arbitrary Lagrangian Eulerian
    - moving boundaries
  - D axisymmetric geometry
  - Comsol Multiphysics<sup>TM</sup>
- Comparison with experiments
- Validation using internal moisture profiles







# **Thermo-hygro-mechanical simulation**



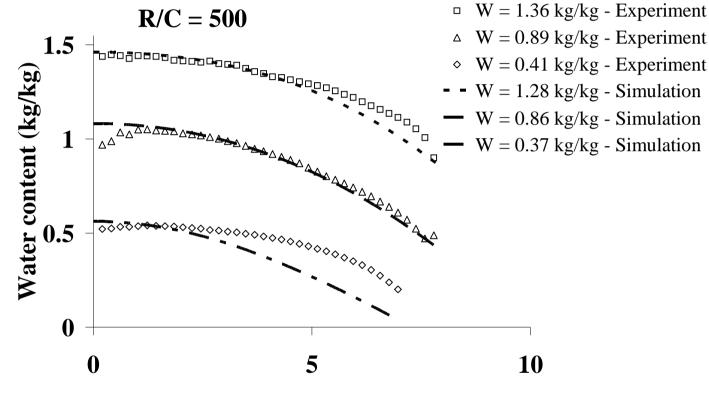






# **Thermo-hygro-mechanical simulation**

Experimental vs simulated moisture profiles



**Distance from center to external border (mm)** 

- Rather good agreement
- Improvement for small water contents











#### **Research context**

Product engineering or product design approach

**Process**  $\Leftrightarrow$  **Microstructure**  $\Leftrightarrow$  **Properties** 



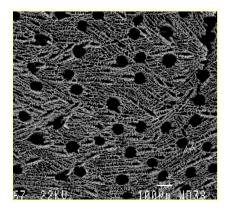
- End user properties, defined by macroscopic properties, depends on microstructure
- Microstructure influenced by production process: synthesis, mixing, dewatering, drying, ...
- Need for multiscale characterization tools
- Key role of X-ray microtomography, coupled with image analysis
- Development of application oriented algorithms

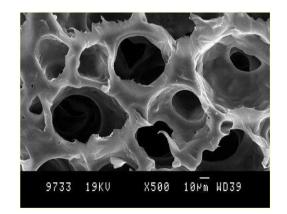




#### **Research context**

- Limitations of classical characterisation techniques
  - Mercury porosimetry: 3.5 nm< d<sub>p</sub><150 μm</li>
  - $\square$  N<sub>2</sub> adsorption-desorption: 2 nm< d<sub>p</sub><50 nm
  - Pycnometry
  - SEM, TEM: destructive, mostly 2D
- Microtomography
  - 3D characterisation
  - Non destructive technique
  - No specific sample preparation
  - $d_p > 2 \mu m$



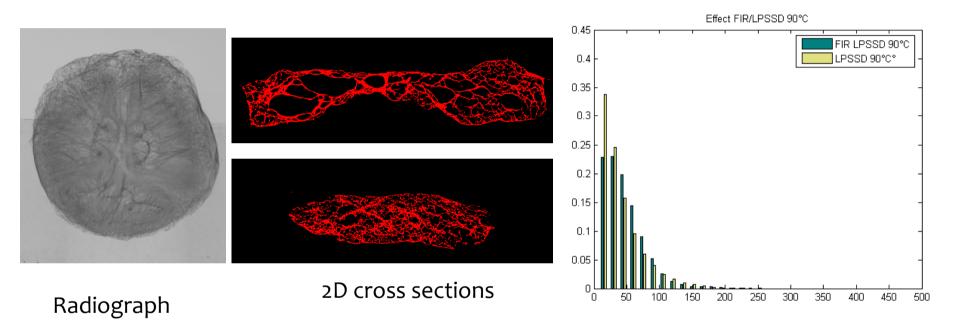






# Impact of drying on product quality

#### Dried bananas



Léonard et al., J. Food Eng. , 85(1), 154-162 (2008)

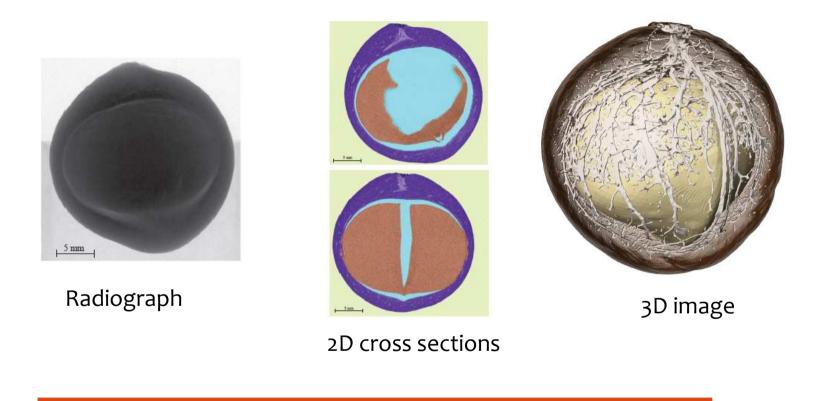
Relation between drying conditions, structure and properties  $\rightarrow$  « crispyness », rehydration, ...





# Impact of drying on product quality

#### Macadamia nuts



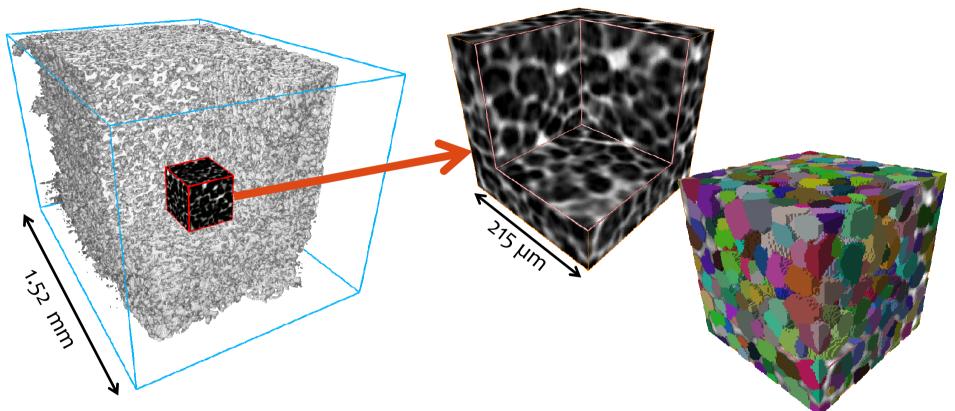
Relation between type, drying, microstructure and conservation





# Characterization of polymer foams

3D microstructure of nanocomposites foams



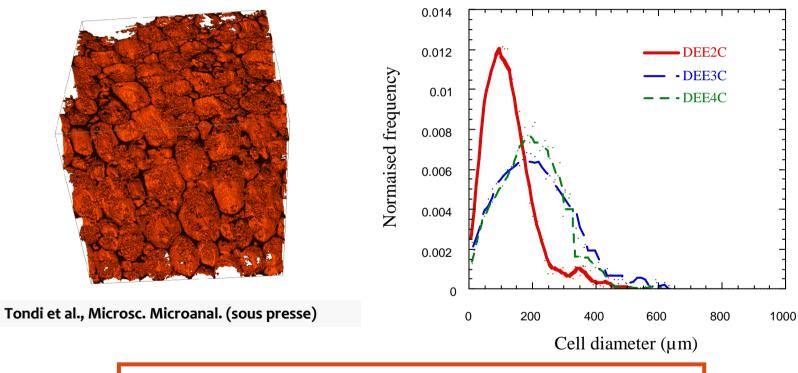
Relation between electrical conductivity and cell morphology





#### Characterization of biobased foams

 Carbon foams based on renewable biomass (Pr. A. Celzard, Nancy)



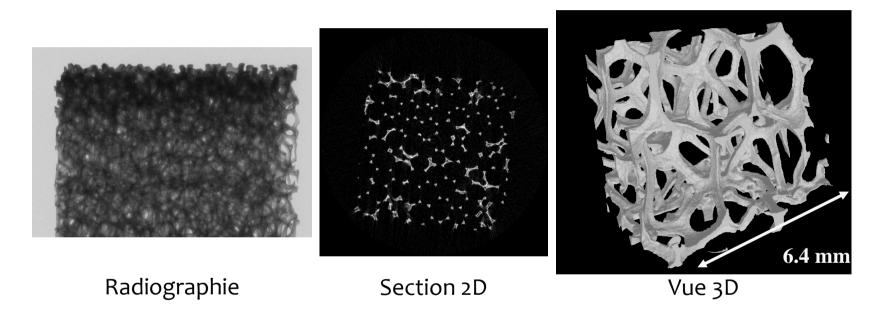
Relation between morphology and synthesis conditions





# Characterization of structured packing

Metallic foams (PhD S. Calvo)



Calvo et al., Chem. Eng. Process., 48(5), 1030-1039 (2009)

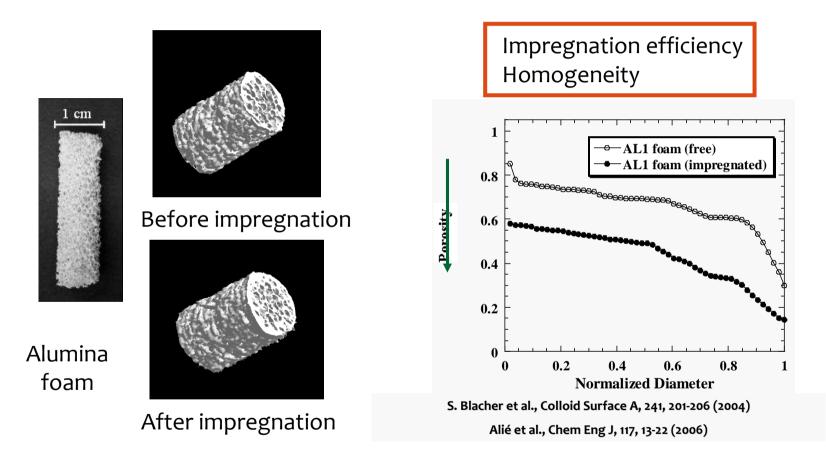
Flow simulation using LBM method





# Impregnation of catalyst

Pd-Ag/SiO<sub>2</sub> xerogel catalyst on alumina foam

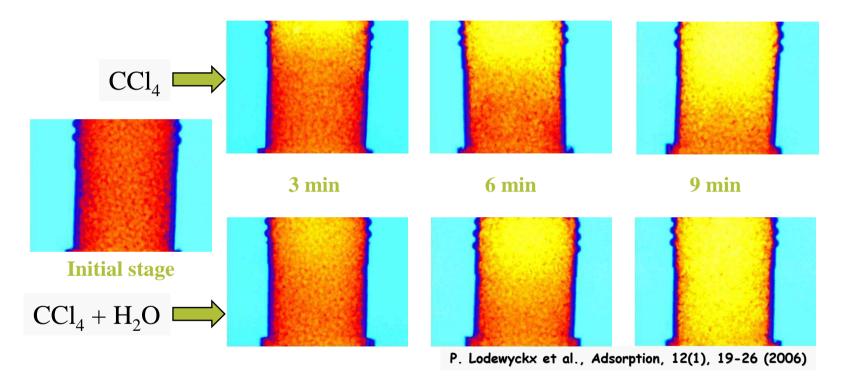






## Follow-up of VOC adsorption

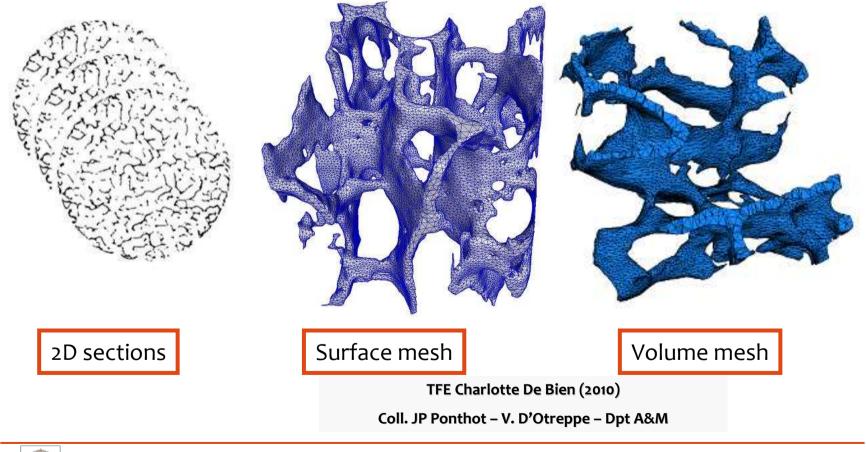
Activated carbon filters





#### **Characterization of biomaterials**

Simulation of compression tests on deer antlers

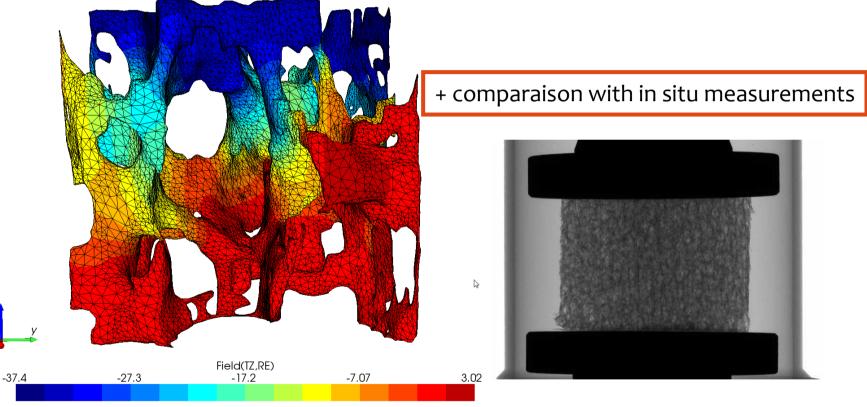




#### **Characterization of biomaterials**

#### Simulation of compression tests on deer antlers

step 30 t=1/1 dt=0.0333333



TFE Charlotte De Bien (2010)

Coll. JP Ponthot – V. D'Otreppe – Dpt A&M





# About LCA





#### LCA: a standardized methodology

 General framework defined by international standards ISO 14040 - 14044

About LCA

- « studies all the environmental aspects and potential impacts associated<sup>End of Life</sup> with all the stages of a product's life from cradle to grave, i.e. from raw material extraction to end of life»
- Product = product, activity, system or process







### LCA: a standardized methodology

- Life cycle includes
  - Raw material extraction
  - Production
  - Transport
  - Packaging
  - Distribution
  - Use
  - Maintenance Repair
  - Reuse or recycling
  - Disposal
- « Cradle to grave » approach

#### **Product Life Cycle**



http://sydney.edu.au/facilities/sustainable\_campus/procurement/index.shtml





# LCA: typical results

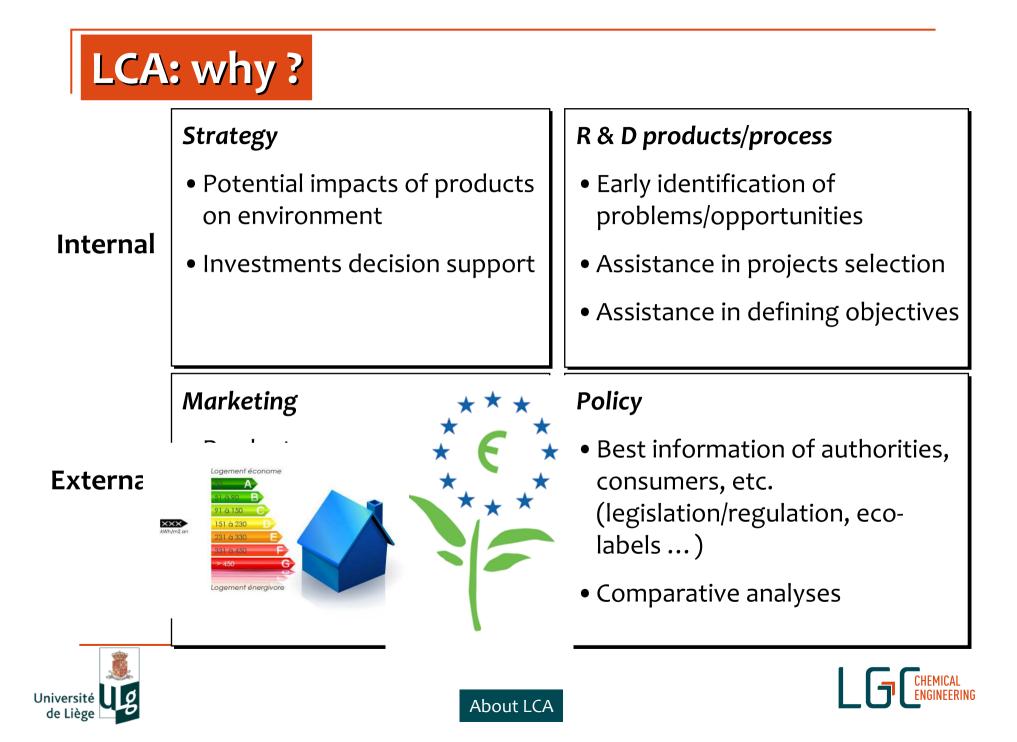
- Life cycle steps 'ranking' following their environnemental impacts
- Identification of susbtances responsible for major environmental impacts

 $CO_2$  footprint = 1 inventory among many others

- Determination of categories with highest environmental impacts
  - □ Human health, climate change, ecotoxicity ...

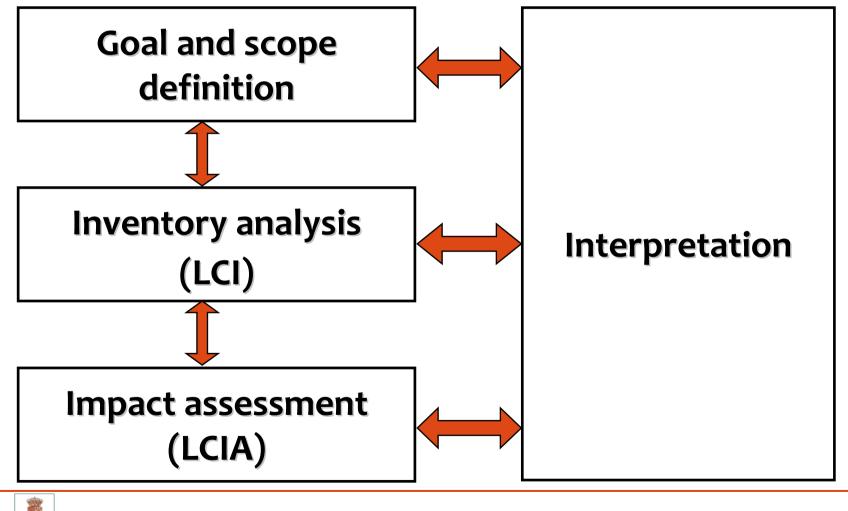






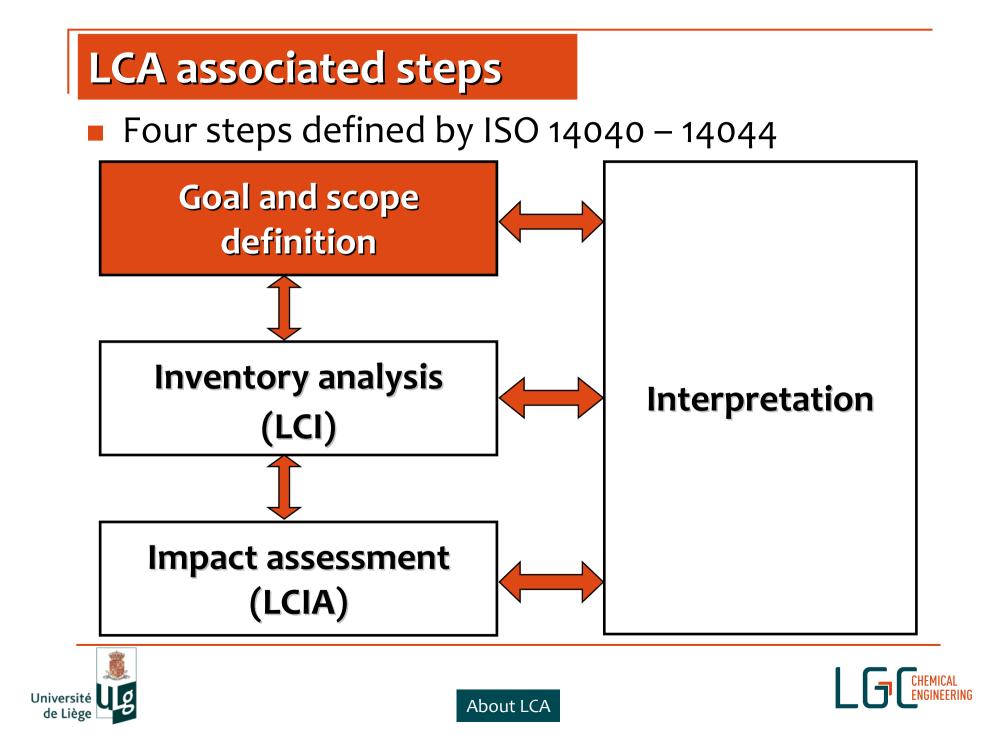


Four steps defined by ISO 14040 – 14044







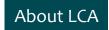


# LCA: goal and scope definition

- Goal of the study
  - Why?
  - For whom ?
  - ...
- Scope
  - Choice of the functional unit
    - Production of 1 kg of yeast
    - Treatment of 1 ton of waste
    - Valorization of 1 ton of biomass





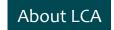


## LCA: goal and scope definition

Scope

- Delimitation of the system boundaries
  - Determination of all the elementary processes to be included in the analysis
- Process tree
- Data quality requirements
- Cut-off rules

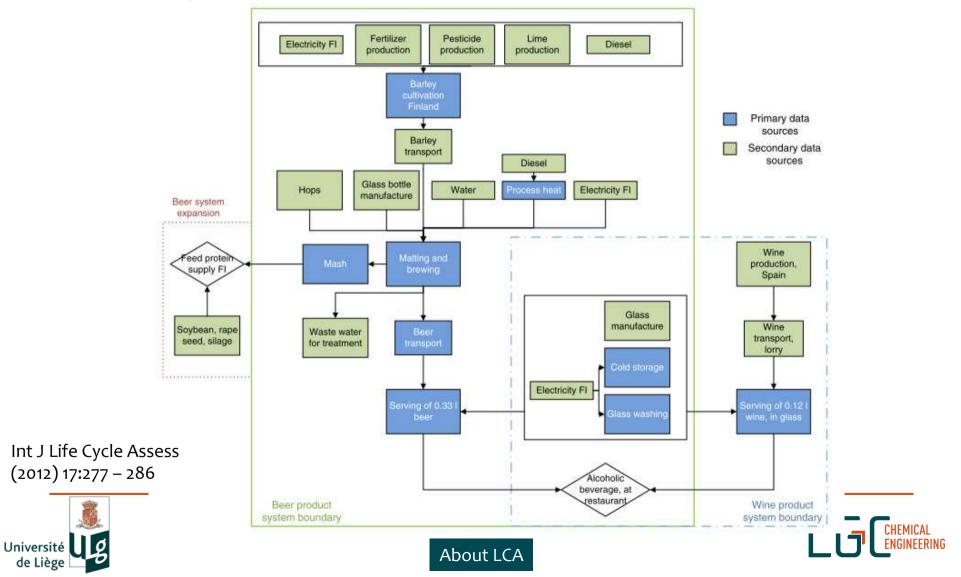


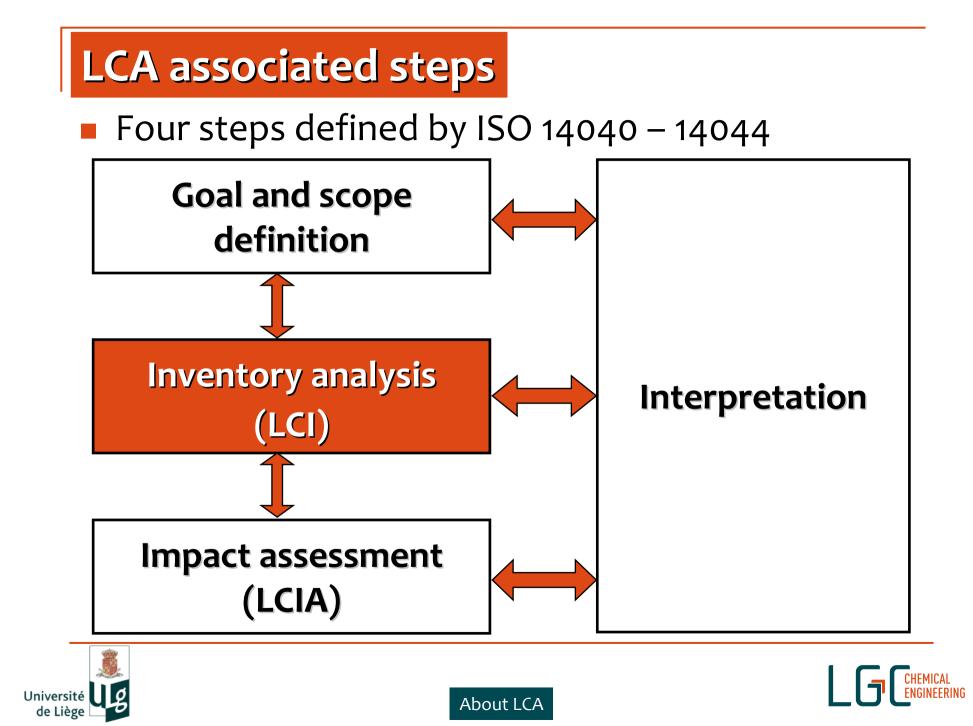




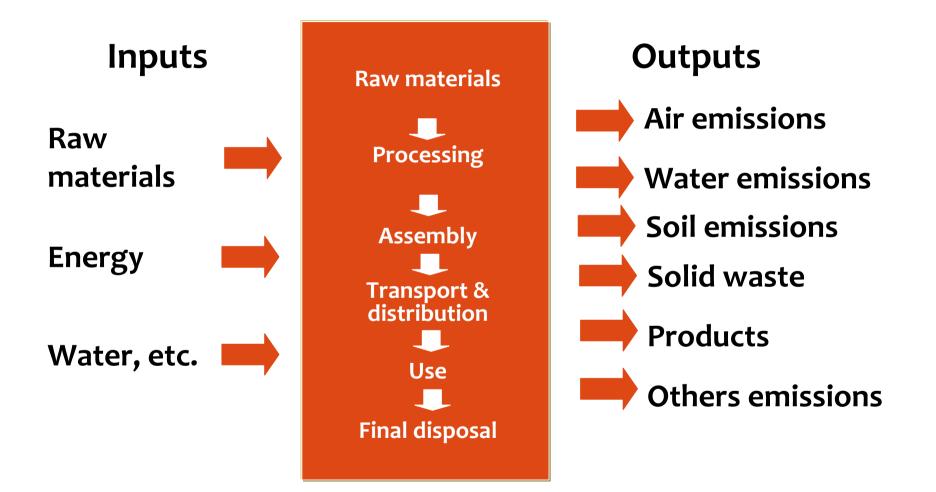
#### LCA: goal and scope definition

#### Example: process tree + boundaries





## **LCA:** Inventory









# **LCA:** Inventory

- Practically ...
  - Commercial databases
  - Data provided by industrials
  - Inter)national databases
  - Scienfitic and technical literature
- But ... data are ofter missing or quality is questioning

About LCA

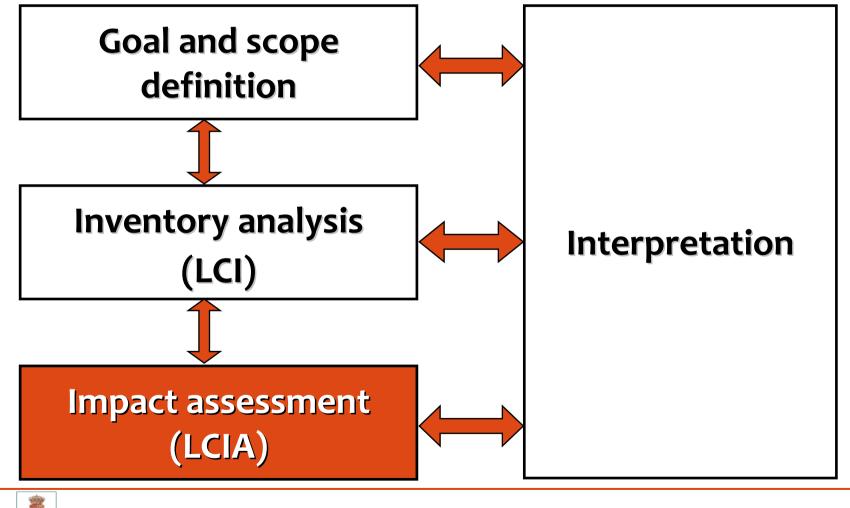
- Process modeling
- Data reconciliation
- □ Knowledge of unit operations, ...







Four steps defined by ISO 14040 – 14044







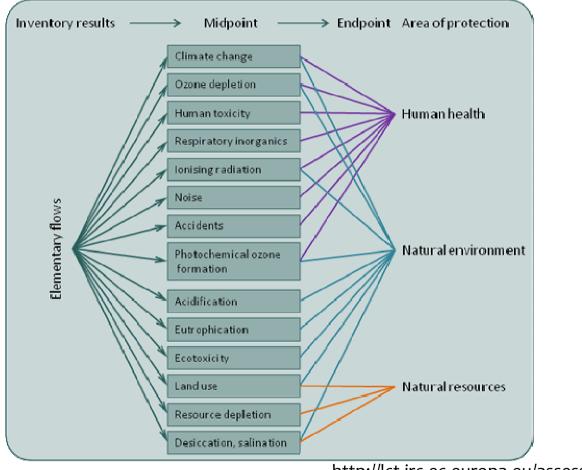
- Estimation of environmental impacts based on the inventory
- Mandatory elements
  - Selection of impact categories, indicators, methods
  - Classification
  - Characterization
- Optional elements
  - Normalisation
  - Grouping
  - Weighting

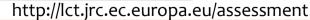




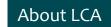


#### Impact categories











#### Impact assessment methods

Method name	Temporal validity	Regional validity	Type of impact category (IC) indicators	Weighting principle
CML 2002	Present state (year 2002)	Global, except for acidification (Europe) and photo-oxidant formation (European trajectory)	Midpoint	No baseline method is proposed
ECO-indicator 99	Present state (year 1999)	Global for the impact categories (IC) climate, ozone depletion and resources. European model for the other IC. Acidification and eutrophication based on Dutch model, land use based on Swiss model.	Midpoint and Endpoint	Three options: Panel method is used for default weights. Monetization and a specific weighting triangle can also be used.
Eco-scarcity	Actual flows reflect 2004 state and critical flows correspond to 2005 political objectives.	Originally developed for Switzerland, but versions for Netherlands, Sweden, Norway, and Japan are also available.	Midpoint-distance to target principle. Endpoints indirectly considered by political targets.	Relative reduction of distance to target by multiplying by the square of the ratio of actual flow and critical flow.
EDIP	Present state (year 2003)	Global	Midpoint	Distance to political targets.
EPS 2000	Present state (year 1999)	Majority global, the largest exception is for Biodiversity where Swedish models are used.	Endpoint effects	Willingness To Pay to avoid changes on safeguard subjects.
ReCiPé	Present state (year 2010)	Europe, but global for climate change, ozone layer and resources.	Combination of midpoint and endpoint methodologies in a consistent way.	Three options: For midpoints a monetization method on the basis of the prevention costs is provided. For endpoints panel weighting is used and monetization on the basis of damage costs can be used.

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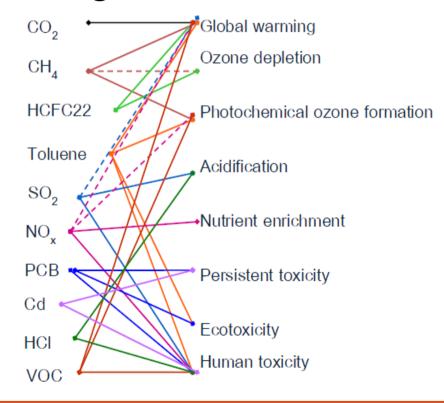


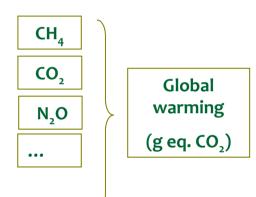


### Classification

Assignment of inventory results to the selected impact categories

About LCA









- Characterization
  - Conversion of LCI results into representative indicators of impact using charaterization factor
  - Substances belonging to the same impact category are expressed in 'equivalent' units

Inventory Data × Characterization Factor = Impact Indicators

. . .

About LCA

 $\begin{array}{ll} \mbox{kg CO}_2/FU \\ \mbox{kg CH}_4/FU & \Longrightarrow \mbox{eq-kg CO}_2/FU \\ \mbox{kg N}_2O/FU \end{array}$ 

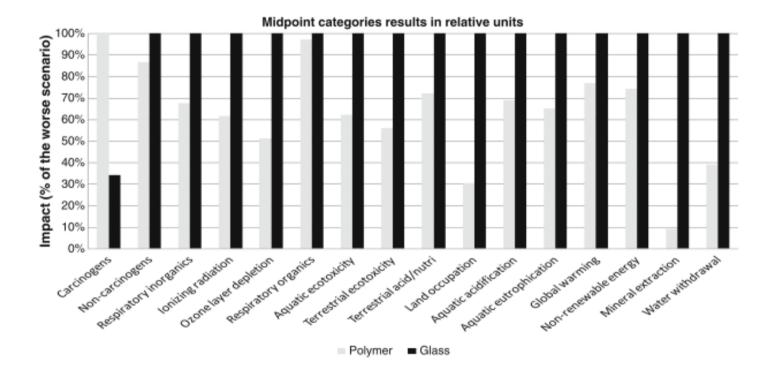
Global warming:eq-lAcidification:eq-lOzone depletion:eq-lFossil fuels:MJ/

eq-kg CO₂/FU eq-kg SO₂/FU eq-kg CFC-11/FU MJ/FU



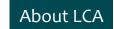


#### Characterization: example - Vials comparison



S. Belboom et al. Int J Life Cycle Assess (2011) 16:159-167





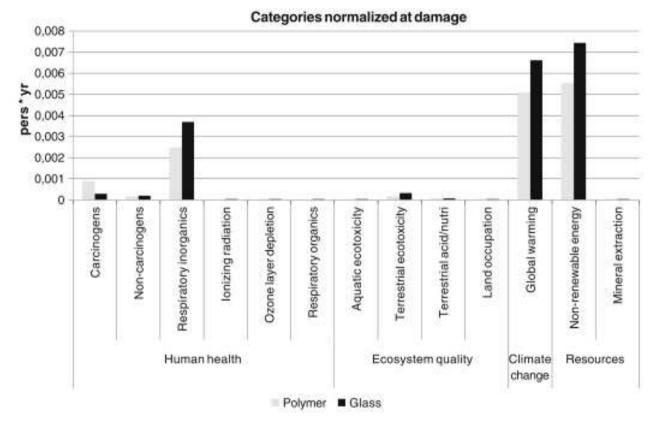


- Normalisation
  - Tool to express impact indicator data in a way allowing to compare among impact categories
  - Indicator normalisation by dividing results by a selected reference value such as global or regional averages
- Grouping
  - Impact categories are sorted and grouped, depending on the chosen impact assessment method





Normalization/Grouping: vials comparison



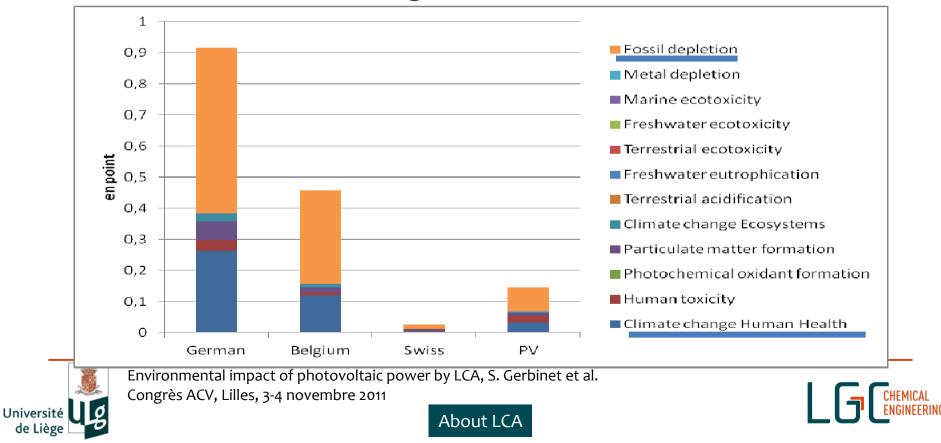
S. Belboom et al. Int J Life Cycle Assess (2011) 16:159-167





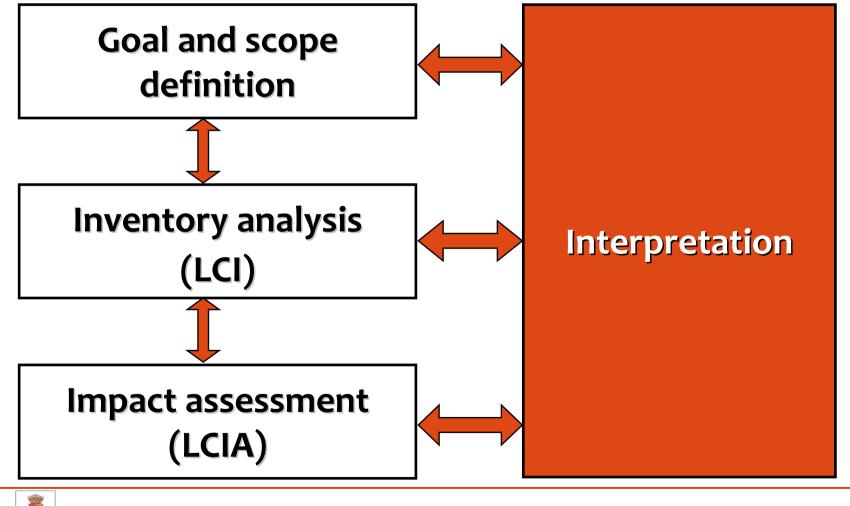


- Weighting
  - Indicator results for different impact categories are converted to a common unit by using factors based on value-choices → "Single score" result





Four steps defined by ISO 14040 – 14044

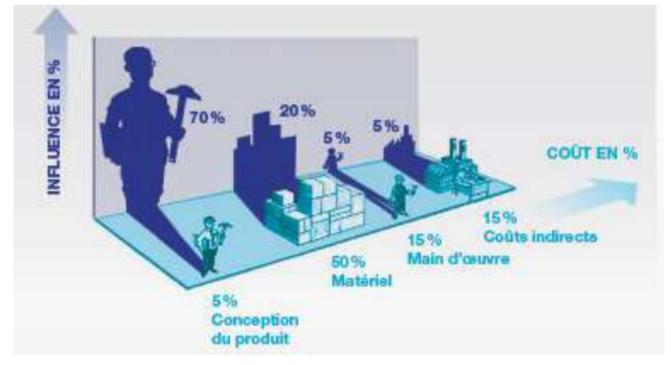






### LCA = way to « ecodesign » new processes

70% of « the environmental impact » already fixed during the design step



Ecodesign = integration of environmental aspects into product or process design with the aim of improving the environmental performance throughout the whole life-cycle

L'écoconception, source d'innovation dans l'approche cycle de vie; l'expérience du Québec, Guy Belletête, Congrès ACV, Lilles, 4/11/2011





About LCA

#### **Expertise in environmental management**

- LCA: more than 10 years of expertise
  - Evaluation of the environmental impact of processes
  - Redaction of environmental declarations
  - Development of databases
  - Academic research + external studies
  - Participation to several regional and European projects
  - • • •







# **Expertise in environmental management**

- LCA: some covered topics
  - Comparison of vehicles
  - Comparison of waste management scenarios
  - Comparison of packaging options
  - Comparison of several ways of renewable heat heat production
  - Comparison of several ways of renewable electricity production
  - Study of biofuels production
  - Study of agro-food by-products valorization ways
  - Impact of water management (whole anthropic water cycle)
  - CO<sub>2</sub> Life cycle inventory of a cement producer
  - Study of several fuel cells configurations
  - LCA training







# **Expertise in environmental management**

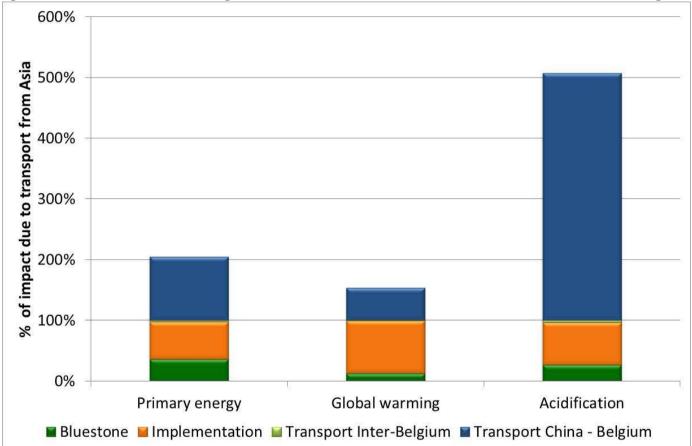
- LCA: current projects
  - WALAID Walloon Region
    - Valorization of the agrofood industry by-products
  - □ SOMABAT FP7
    - Development of novel SOlid MAterials for high power Li polymer BATteries
      - □ LCA of the developped batteries + recyclability
  - Pierres et marbres de Wallonie
    - Realisation of environmental product declarations (FDES)
      - Blue stone, sand stone, ...





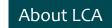
# **Bluestone vs. Chinese stone**

#### Impact of transport on environmental impact



LCA as decision tool for sustainable choices in mineral materials field: environmental declarations of Belgian products and their foreign equivalents, S. Belboom et al. Congrès ACV, Lilles, 3-4 novembre 2011

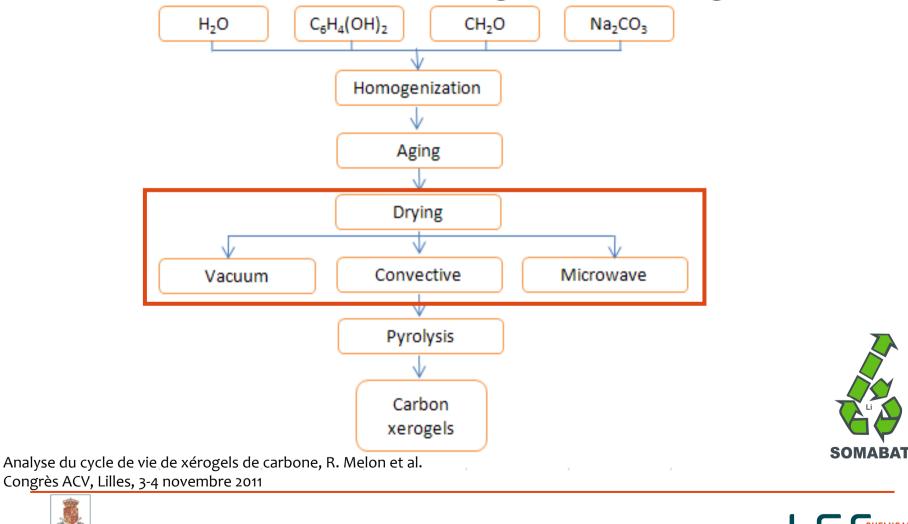






# **Comparison of drying technologies**

Production of carbon xerogels: ecodesign approach

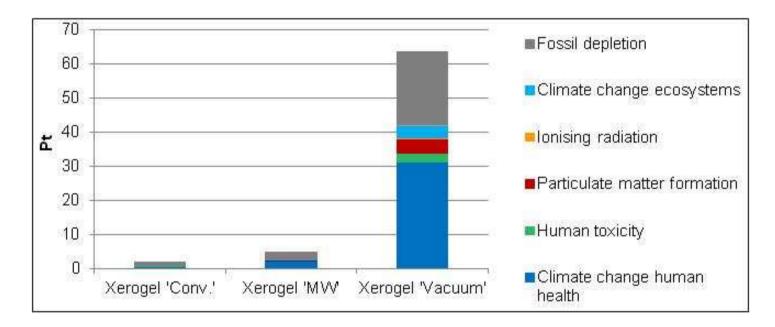






# Comparison of drying technologies

#### Weighted results



Best environmental choice = convective drying



Analyse du cycle de vie de xérogels de carbone, R. Melon et al. Congrès ACV, Lilles, 3-4 novembre 2011







#### **Comparison of waste treatment technologies**

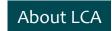
- Before 1970: wild landfilling
- From 1990 until 2009: waste grinding and sorting



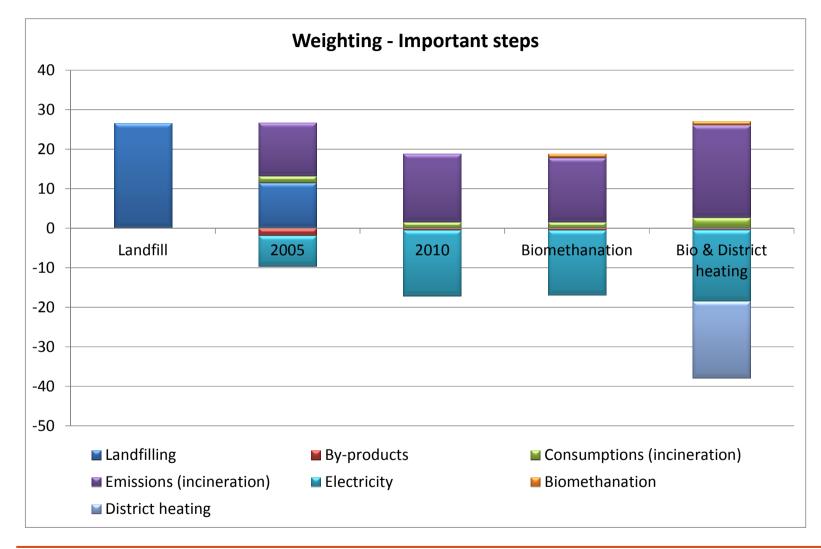
- From 2009 until now: incineration of the whole waste fraction
- Short term project: methanation of the biodegradable fraction after collect and sorting







#### **Comparison of waste treatment technologies**







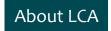


# LCA ... a nice tool but ...

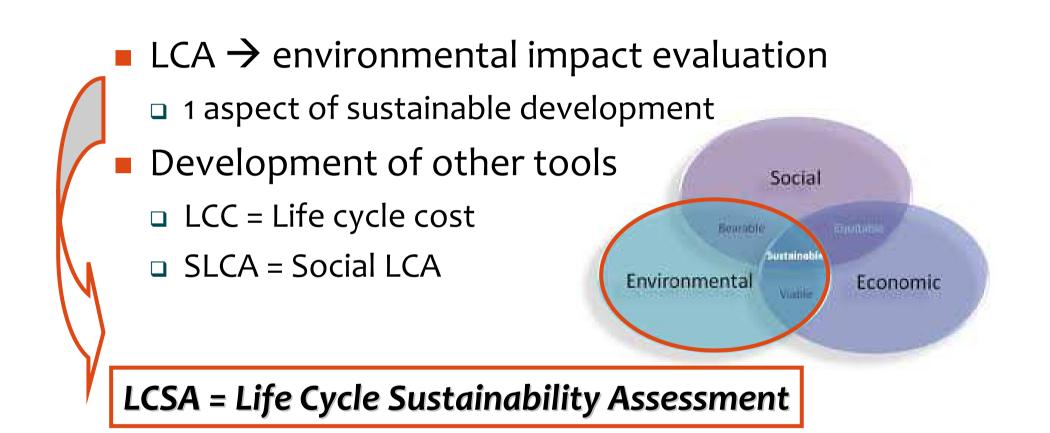
- Difficult to compare different environmental impacts
- Impacts related to equipments and infrastructure usually not taken into account
- Difficult to formulate « easy to understand » conclusions for non-experts
- Need to prevent for abusive and « out of context » use of results
- Clear statement of results validity (uncertainties, limit of the study, hypothesis, ...)
- Availability and quality of data
- Need for more transparency about some subjective choices
- Need for new assessments methods: water use, land use/land use change, ...



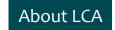




# LCA ... a nice tool but ...









# To conclude ... Thanks to the « 3 teams » !





# The team

- Drying
  - Prof. Michel Crine
  - Thierry Salmon (industrial engineer)
  - Dr Lyes Bennamoun (FRS-FNRS postdoc)
  - Laurent Fraikin (PhD student)
  - Yvon bert PAMBOU (PhD student)
- X-ray microtomography
  - Prof. Dominique Toye
  - Dr Erwan Plougonven (FRS-FNRS postdoc)

Intro to LGC

Charlotte De Bien (FRIA PhD student)





# The team

- LCA
  - Sandra Belboom (PhD student)
  - Saïcha Gerbinet (PhD student)
  - Raphaëlle Melon (Industrial engineer)
  - Robert Renzoni (Senior researcher)





# Dank u voor uw aandacht !

# Vragen?



