



**ValBran**

# Valorisation of wheat bran into surfactant molecules

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Representing the ValBran consortium

[www.valbran.eu](http://www.valbran.eu)

Facebook page: @Valbiom @interregFranceWallonieVlaanderen #ValBran  
LinkedIn & Twitter: ValBiom asbl #ValBran

# Interreg France-Wallonie-Vlaanderen



62.000 km<sup>2</sup>  
10.800.000 habitants/inwoners



## ONDERZOEK EN INNOVATIE RECHERCHE ET INNOVATION

1. Versterken van het onderzoek en de innovatie van de grensoverschrijdende zone in de strategische sectoren en de sectoren met een sterke complementariteit | Accroissement de la recherche et de l'innovation de la zone transfrontalière dans les secteurs stratégiques et les secteurs à forte complémentarité
2. Grottere overdracht en verspreiding van goede praktijken in de strategische sectoren en de sectoren met een sterke complementariteit in de grensoverschrijdende zone | Accroissement du transfert et de la diffusion des bonnes pratiques innovante dans les secteurs stratégiques et à forte complémentarité de la zone transfrontalière



Projet soutenu par



Recherche et innovation

Plus d'infos

[www.interreg-fvwl.eu](http://www.interreg-fvwl.eu)  
@InterregFWVL

Ghent, Bio Base Europe Pilot Plant - 7.11.2019

Avec le soutien du Fonds européen de développement régional

# Alkyl glycosides and sugar esters, non-ionic surfactants of interest

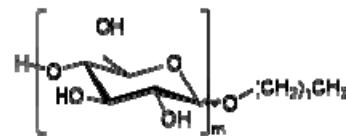
Non-ionic surfactants ≈ 50 % of the total surfactant market in Europe

Growing interest for surfactants from renewable resources:  
biodegradability, non toxic for humans and environment

## Alkyl polyglycosides (APG)

World market: 100,000 T/year

Emulsifiers, foaming agents, wetting agents for  
cosmetics, detergents, phytosanitary

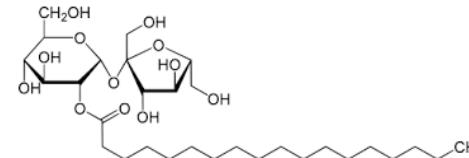


Appyclean™ (Wheatoleo),  
Oramix™, Montanov™  
(Seppic), ...

## Sugar esters

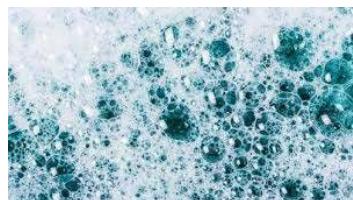
World market: 10,000 T/year

Emulsifiers for cosmetics and food



CrodestaTM (Croda),  
products from Sisterna, ...

**Chemically produced:** limited degree of polymerization (DP) for the glycon part which  
impacts hydrophilic-lipophilic balance, generation of undesirable molecules



# Alkyl glycosides and sugar esters, non-ionic surfactants of interest

## Wheat bran valorisation into surfactants



- **Wheat bran**, agricultural co-product from milling industries and 1G bioethanol industries
- Main use: **food (fibers)** and **feed**

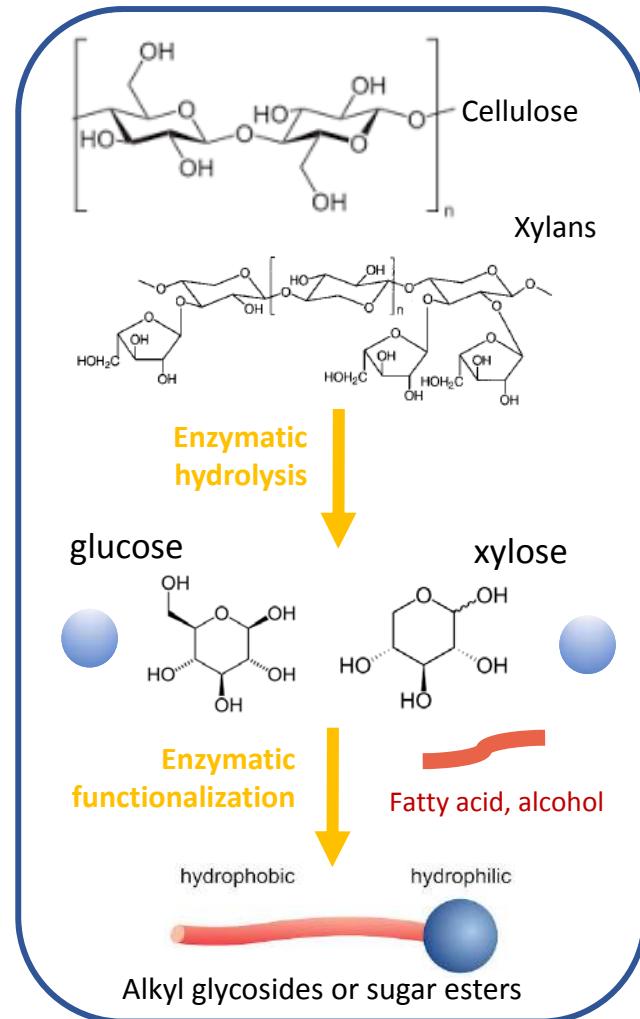
### Enzymatic processes environmentally friendly

- Cellulose and xylans fractionation
- Functionalization of sugars from fractionation

### Surfactants for high added-value applications

Cosmetics, food...

Wheat bran residues  
(+ cellulases-hemicellulases): feed



# Partners



# Overview of the project



# Enzymatic synthesis of alkyl xylosides

## Enzymatic transglycosylation

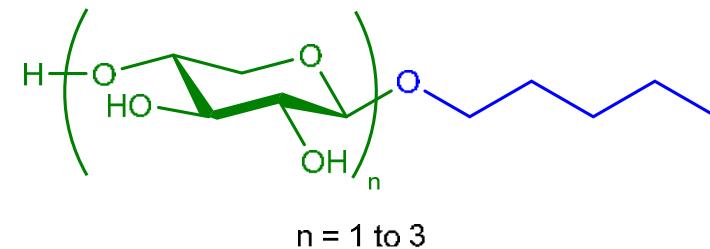
Wheat bran

No pretreatment



xylanase  
pentan-1-ol /  
 $\text{H}_2\text{O}$   
48h, 50°C

Example: synthesis of pentyl  $\beta$ -D-xylosides



# Enzymatic synthesis of alkyl xylosides

- Synthesis efficient with wheat bran from Biowanze (Wanze, Belgium) and from ADM (Pomacle Bazancourt, France)
- Optimization of the transglycosylation reaction conditions:
- Petyl  $\beta$ -D-xylosides : 4.9 g/L  
450 mg pentyl  $\beta$ -D-xylosides / g xylans present in wheat bran  
→ *equivalent to transglycosylation performed from commercial xylans*  
Ochs et al. Green Chem., 2011, 13, 2380

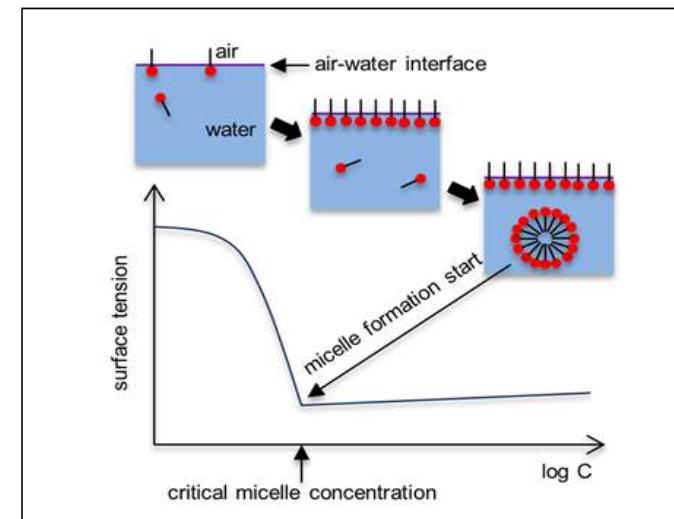
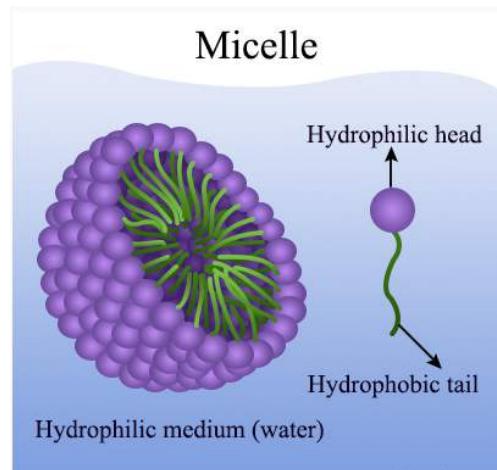
## Enzymatic synthesis of alkyl xylosides

- Up-scaling of the reaction in a 2 liters reactor  
→ same yields of transglycosylation
- Reaction efficient for the synthesis of alkyl  $\beta$ -D-xylosides with C chain lengths from C3 to C10 (propan-1-ol to decan-1-ol)

# Properties of the pentyl xylosides

## Surface-active properties

- Capacity to decrease the surface tension ( $\gamma_{CAC}$ )
- Aggregation behaviour: Critical aggregation concentration (CAC)

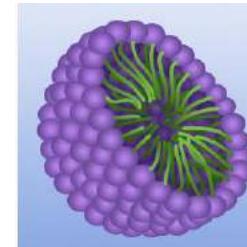


$(CAC, \gamma_{CAC})$

# Properties of the pentyl xylosides

## Surface-active properties - Results

Pentyl $\beta$ -D-xylosides mixture	CAC ( $\text{mg} \cdot \text{L}^{-1}$ )	$\gamma_{\text{CAC}}$ (mN/m)
From WB 1	3089	59
From WB 2	2150	39



Literature (Bouxin *et al.*, 2010)

Octyl  $\alpha/\beta$ -D-xylosides DP1 : CMC = 950  $\text{mg} \cdot \text{L}^{-1}$

- Poor surface-active properties – longer alkyl chain (C8 or C10)
- But potential hydrotrope properties (under investigation)

## Enzymatic synthesis of sugar esters

1<sup>st</sup> step : production of glucose and xylose hydrolysates from wheat bran

Wheat bran  
No pretreatment



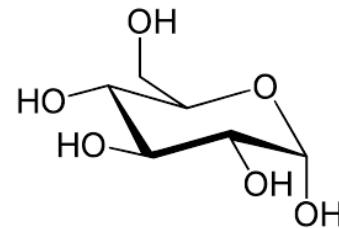
Wheat bran from [BIOWANZE](#)  
(Wanze, BE) and from [ADM](#)  
(Pomacle-Bazancourt, FR)

Enzymatic hydrolysis

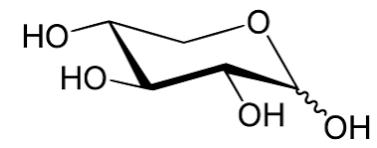
H<sub>2</sub>O, 50°C

No additives

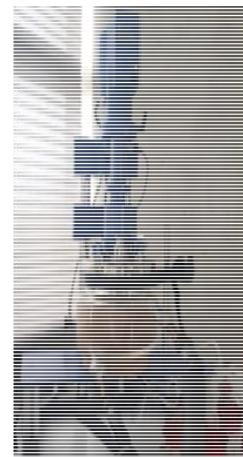
D-glucose



D-xylose



According to enzyme loading and reaction duration:  
**50 to 100%** of glucose and xylose released



Hydrolysate rich in glucose and xylose

Starch and wheat bran residues

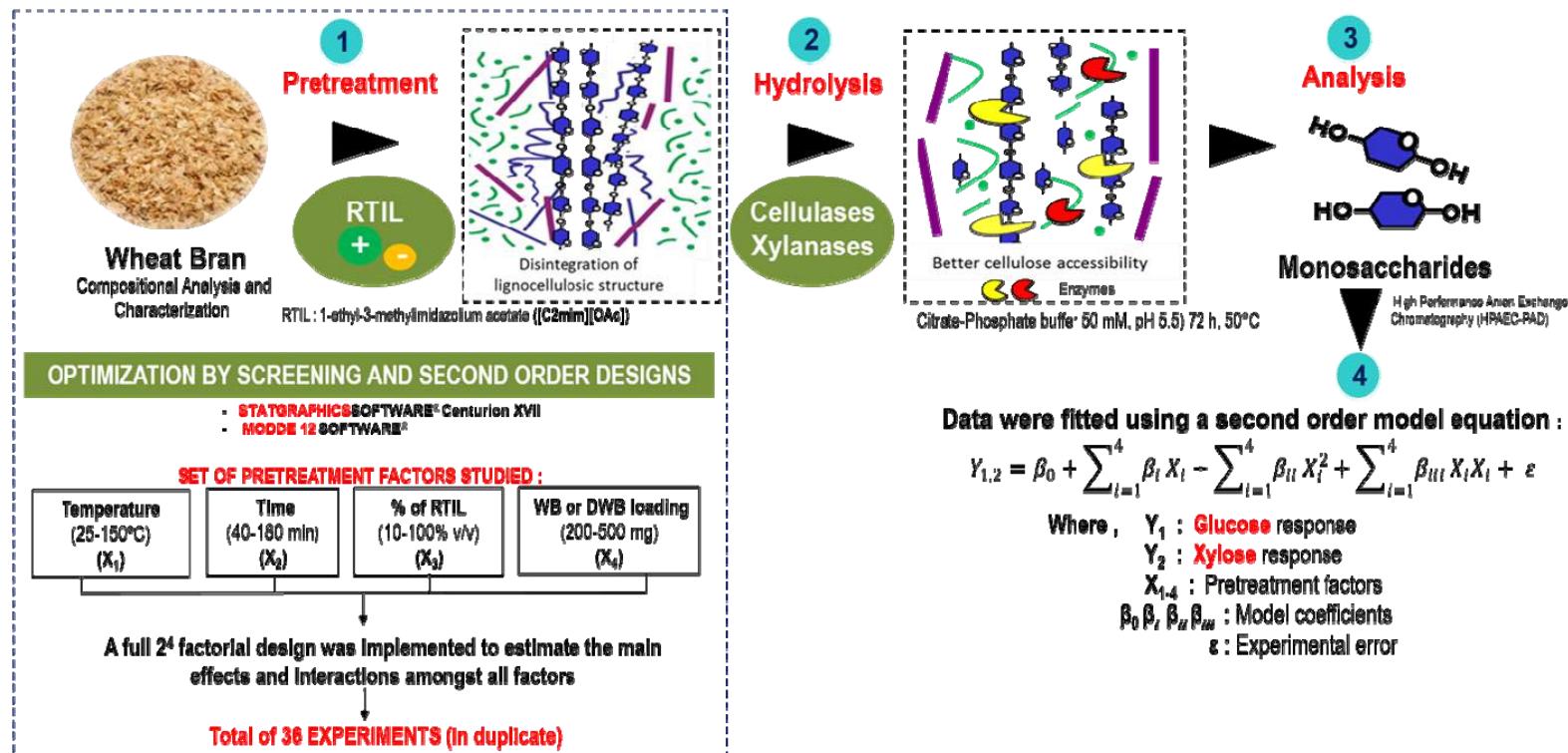
# Enzymatic synthesis of sugar esters

1<sup>st</sup> step : production of glucose and xylose hydrolysates from wheat bran

Scientific and technological lock: recalcitrance properties of wheat bran



Prescribed solution: Room Temperature Ionic liquid (RTIL) pretreatment prior enzymatic hydrolysis

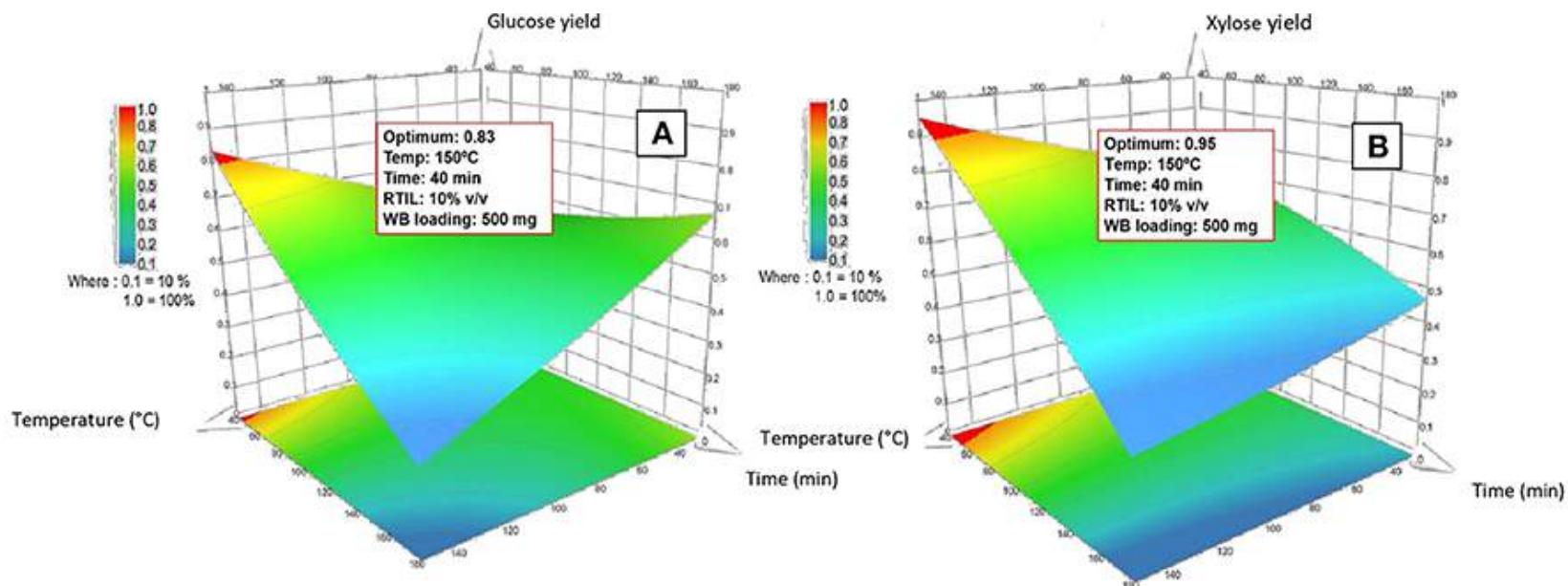


Optimization of conditions pretreatment by PLS-Surface Response Design

# Enzymatic synthesis of sugar esters

1<sup>st</sup> step : production of glucose and xylose hydrolysates from wheat bran

Response surface plots of sugar (A: glucose, B: xylose) release after enzymatic hydrolysis



Optimal conditions of pretreatment: diluted-[C2mim][OAc] in water (10 % v/v) at 150 °C for 40 min

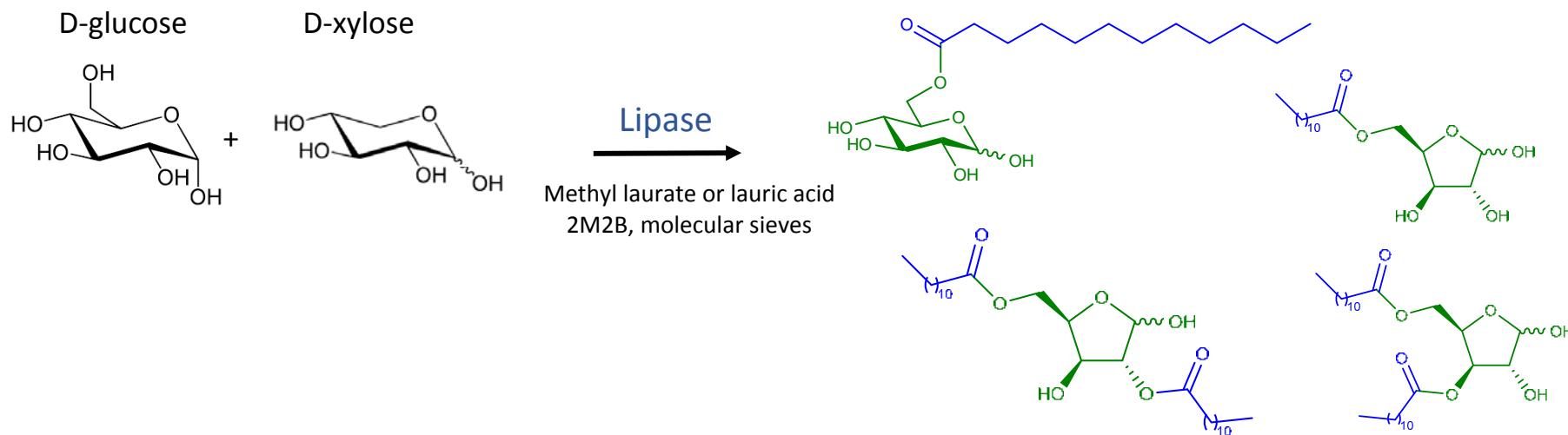
Competitive yields achieved:  $Y_{\text{glucose}} = 83\%$  and  $Y_{\text{xylose}} = 95\%$

Araya-Farias *et al.* (2019). Frontiers in Chemistry. 7:585

# Enzymatic synthesis of sugar esters

2<sup>nd</sup> step : production of sugar esters from glucose and xylose hydrolysates

Example: synthesis of laurate glucose and xylose esters



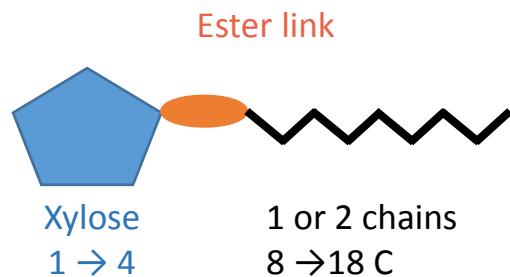
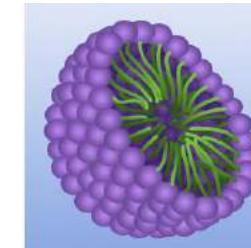
- Sugars conversion: 80-100%
- Modulation of **glc esters and xyl esters ratios and of mono- and diesters ratios** according to D-glc/D-xyl ratio (hydrolysate) and reaction conditions
- (Trans)esterification efficient with fatty acids and fatty acids esters with **C chain lenght from C8 to C18**

Méline *et al.* Enz. Microbial. Technol. 2018

# Properties of the sugar esters

## Surface-active properties

- Structure-activity relationships of purified molecules from (trans)esterification reactions



XC8	Xylose octanoate monoester
X(C8)2	Xylose octanoate diester
XC12	Xylose laurate monoester
XC14	Xylose myristate monoester
XC18	Xylose stearate monoester
X2C12	Di-Xylose laurate monoester
X3C12	Tri-Xylose laurate monoester
X4C12	Tetra-Xylose laurate monoester
XyC12	Xylo-oligosaccharides laurate monoesters

Number of alkyl chains

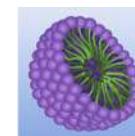
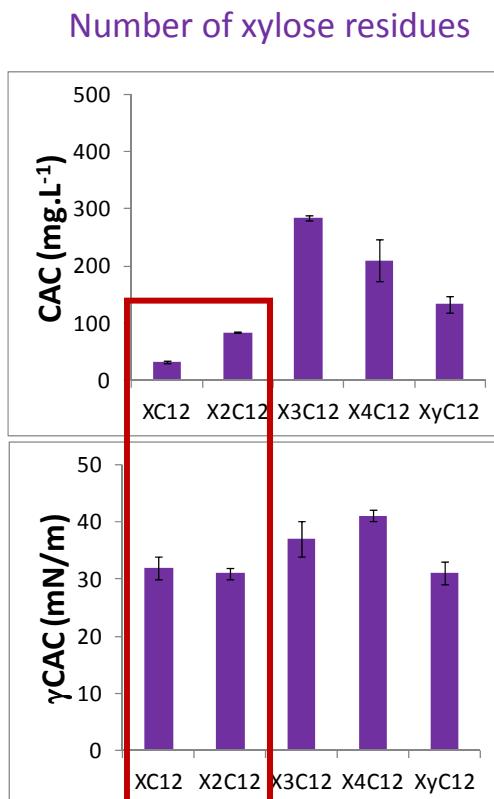
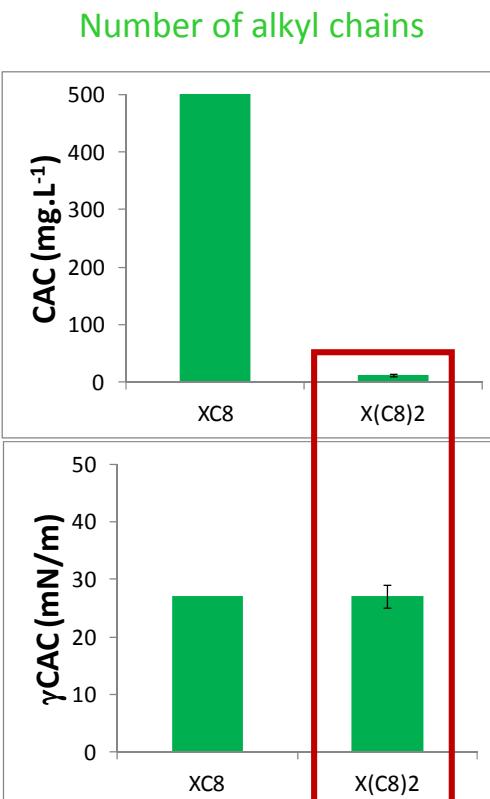
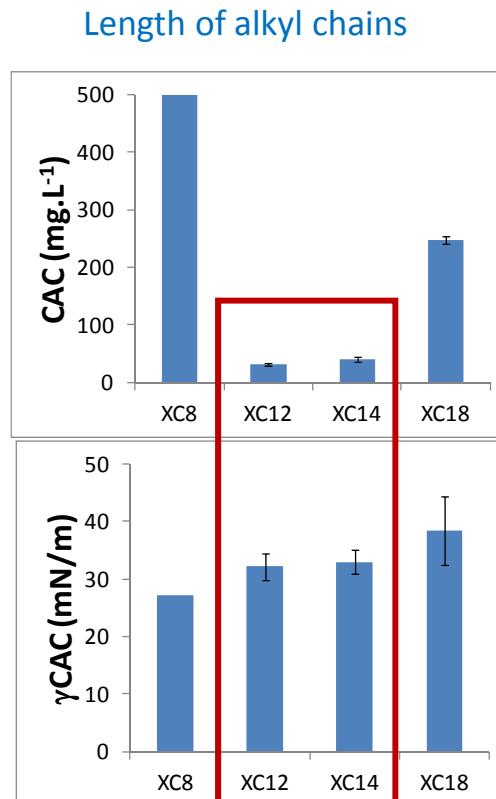
Length of alkyl chains

Number of xylose residues

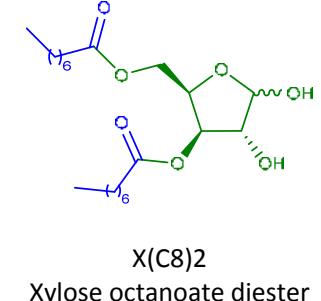
# Properties of the sugar esters

## Surface-active properties

### ➤ Structure-activity relationships of purified molecules- Results



The best candidate:



Middle chain length

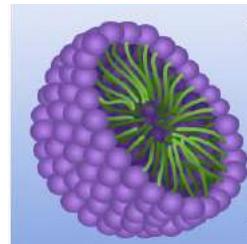
Two alkyl chains

Limited number of xylose residues

## Properties of the sugar esters

### Surface-active properties

- Structure-activity relationships of **mixtures from (trans)esterification reactions obtained from wheat bran**

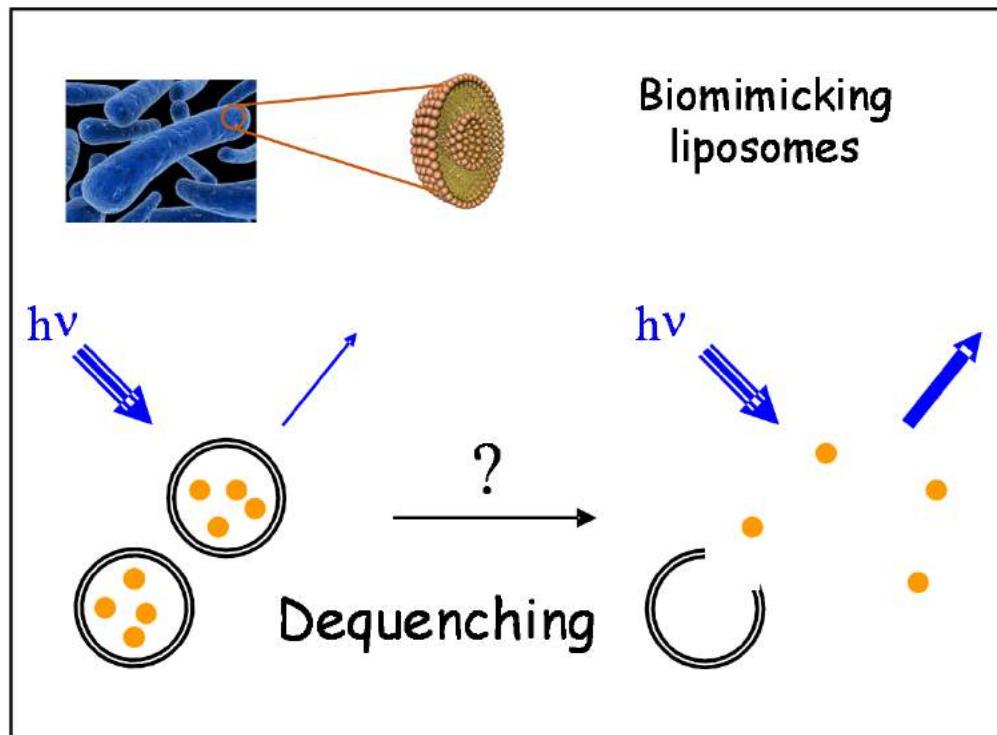


D-glc and D-xyl lauryl esters mixture	CAC ( $\text{mg} \cdot \text{L}^{-1}$ )	$\gamma_{\text{CAC}}$ (mN/m)
Mixture with mono-esters mainly	44.4	24.4
Mixture with di-esters mainly	48.3	27.5

# Properties of the sugar esters

## Potential as antibacterial agent

- Permeabilization of bacterial plasma membrane



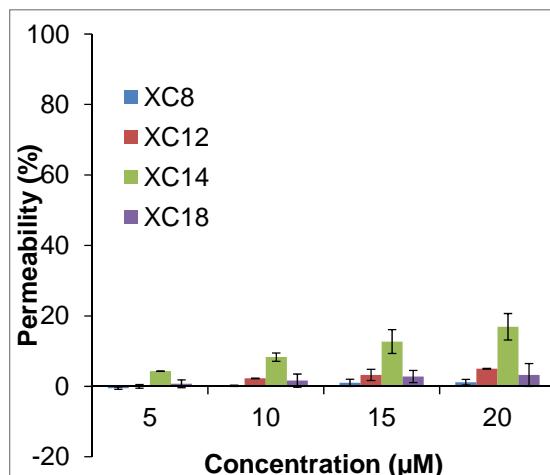
# Properties of the sugar esters

## Potential as antibacterial agent

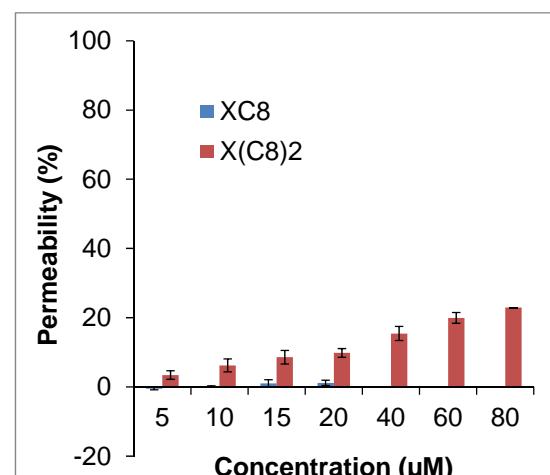
- Permeabilization of bacterial plasma membrane - Results



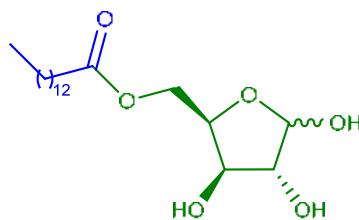
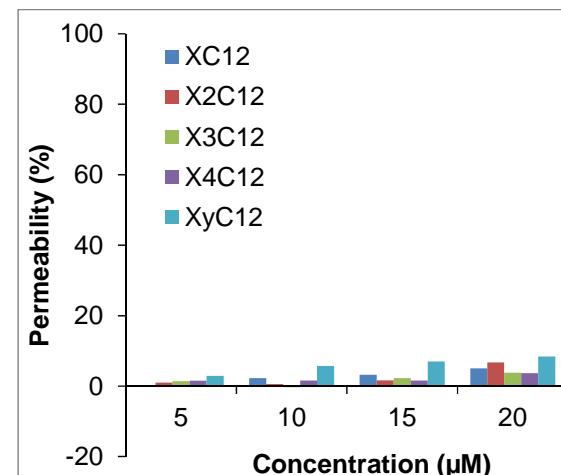
Length of alkyl chains



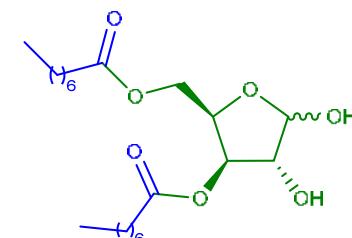
Number of alkyl chains



Number of xylose residues



- Moderate permeability of bacterial membranes
- The best candidates : XC14 and X(C8)2



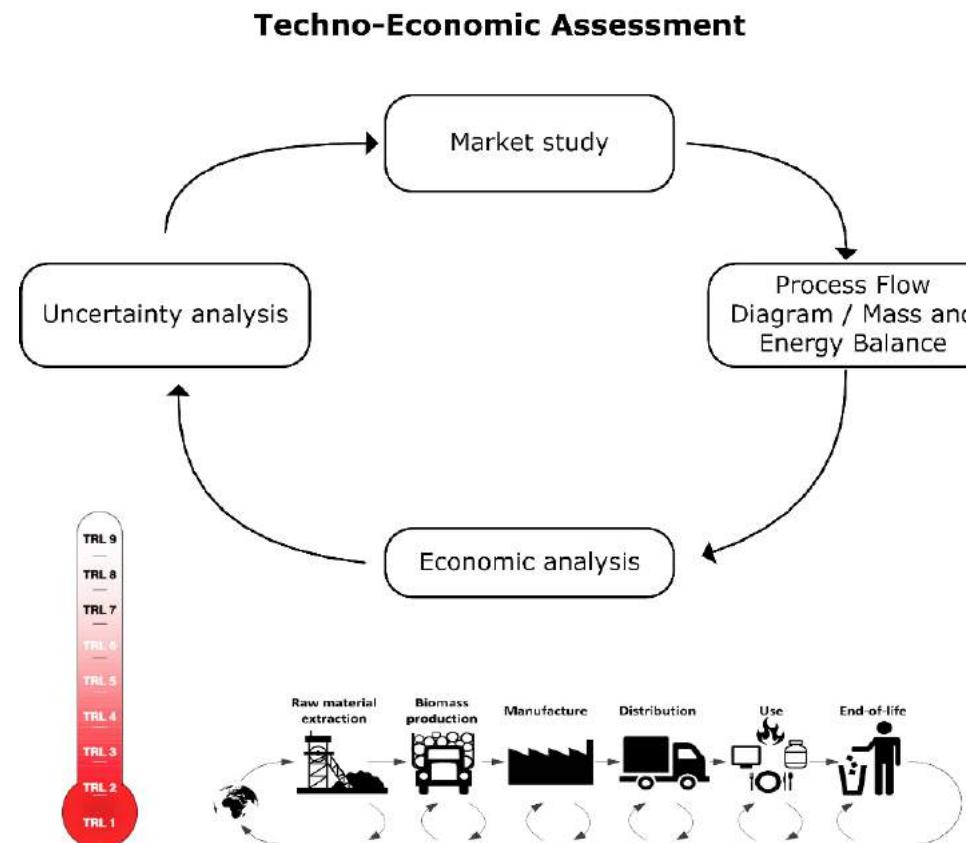


# TEA - TECHNO-ECONOMIC ASSESSMENT

by Yamini Satyawali

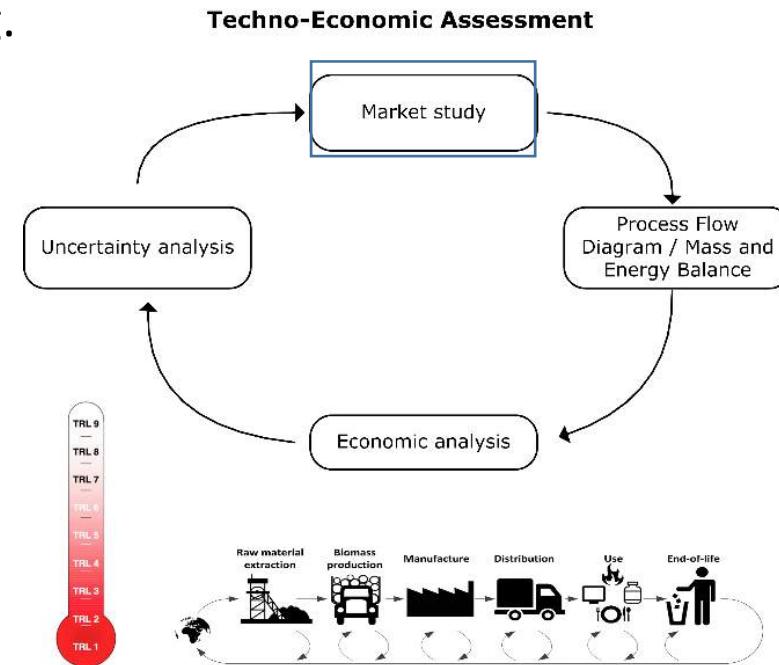
VITO, Flemish Institute for Technological Research

## Techno-economic assessment

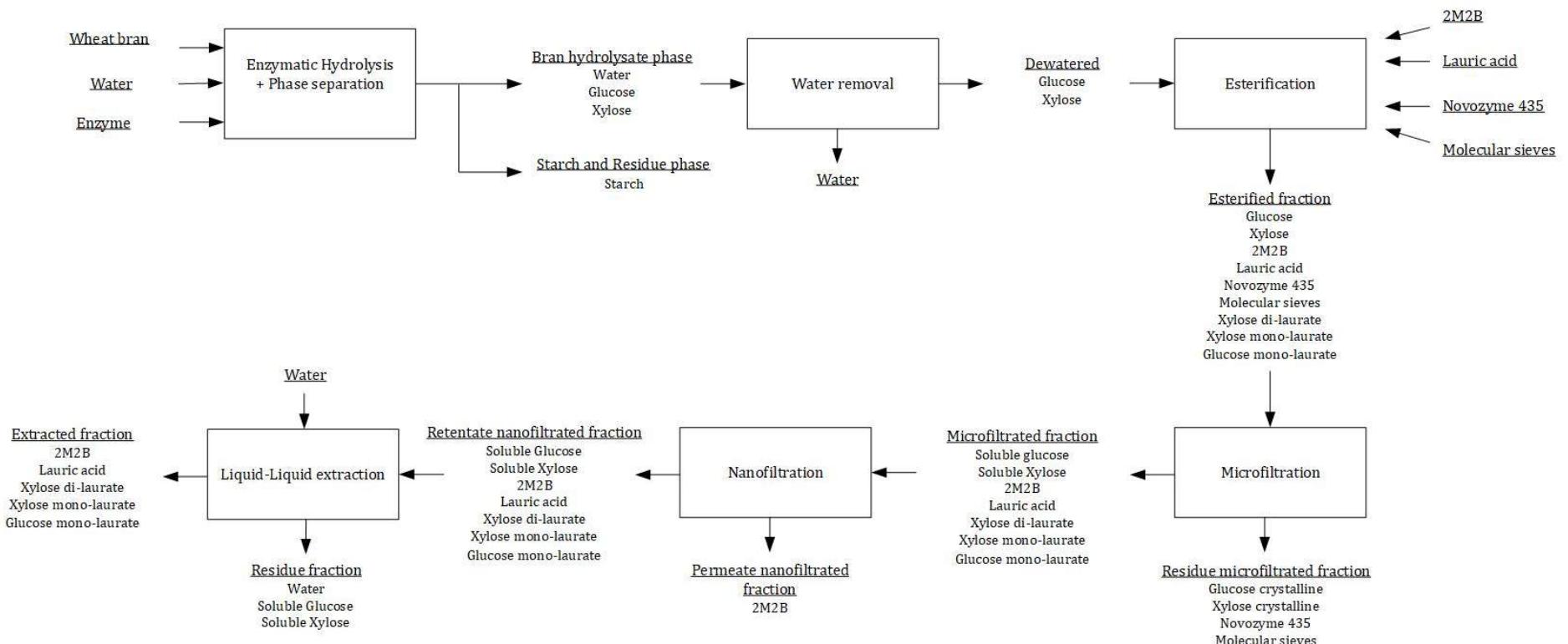


## Techno-economic assessment – Market study

- Market study collects information on e.g.
  - Market demand and growth
  - Product characteristics
  - Prices
  - Competitors
  - Legal aspects
  - ...



## Techno-economic assessment – Technical assessment



## Techno-economic assessment – Economic assessment

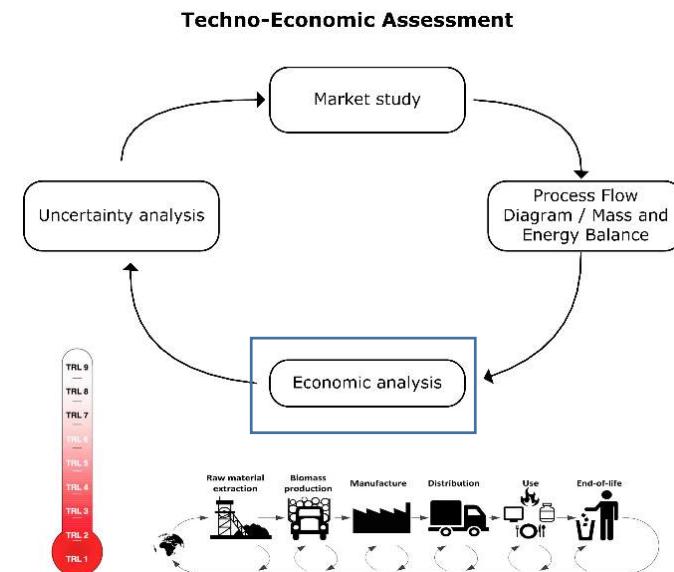
### ■ Economic Assessment

Calculation of:

- CAPEX
  - OPEX
  - Revenues
- Integrated with Mass and Energy balance

Result:

- Production cost
- Investment criteria such as Net Present Value, Internal Rate of Return and (Discounted) Payback Period

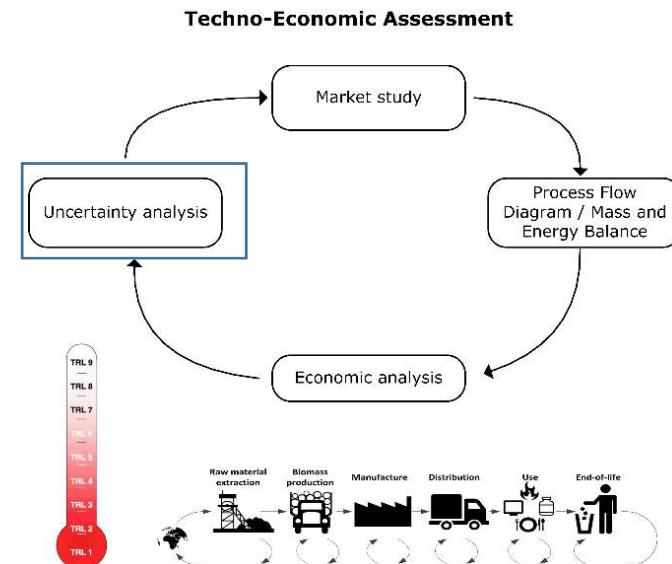


## Techno-economic assessment – Uncertainty

- **Uncertainty analysis**
  - Many uncertainties in technical and economic parameters
  - Monte Carlo assessment to see which parameters have the highest impact on e.g. the production cost or NPV

→ For the most important parameters: Can we improve them?

- If yes, how would it impact the viability?
- If no, do we have alternatives available?



## Bio-based surfactants from ValBran: Opportunities for testing applications

Under progress: technological transfer - WP6

- Economical feasibility (TEA)
- Environmental impact (LCA)



Surfactants from ValBran can be provided to companies for evaluation

Contact: caroline.remond@univ-reims.fr

**Janvier 2017 – Décembre 2020**  
**Januari 2017 – December 2020**

**Coût total : 1.745.826,28 € | Financement FEDER : 872.913,12 €**  
**Totale kost : 1.745.826,28 € | EFRO-financiering : 872.913,12 €**

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Avec le soutien du Fonds européen de développement régional, de la Région Grand Est, de la Wallonie et de la Province West-Vlaanderen.  
Met steun van het Europees Fonds voor Regionale Ontwikkeling (EFRO), Grand Est, Wallonië en Vlaanderen.



Merci pour votre attention  
Dank u wel voor uw aandacht

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