

# Mild oxidative organosolv pretreatment of lignocelullosic biomass residues for high added value chemicals and food additives via fermentation processes

# <u>Konstantinos G. Kalogiannis</u><sup>1</sup>, C.M. Michailof<sup>1</sup>, A.A. Lappas<sup>1</sup>, A. Chalima, A. Karnaouri<sup>2</sup>, E. Topakas<sup>2</sup>,

<sup>1</sup>Chemical Process and Energy Resources Institute (CPERI), Centre for Research and Technology Hellas (CERTH) <sup>2</sup>Biotechnology Laboratory, School of Chemical Engineering, National Technical University of Athens

\*kkalogia@cperi.certh.gr

Novel Conversion Technologies of Waste Biomass to Food additives and Fine Chemicals

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## People behind this work

Chemical Process and Energy Resources Institute (CPERI), Centre for Research and Technology Hellas (CERTH)





Dr. Michailof Dr. Lappas Biotechnology Laboratory, School of Chemical Engineering, National Technical University of Athens



Dr. Karnaouri



Ms Chalima, PhD student



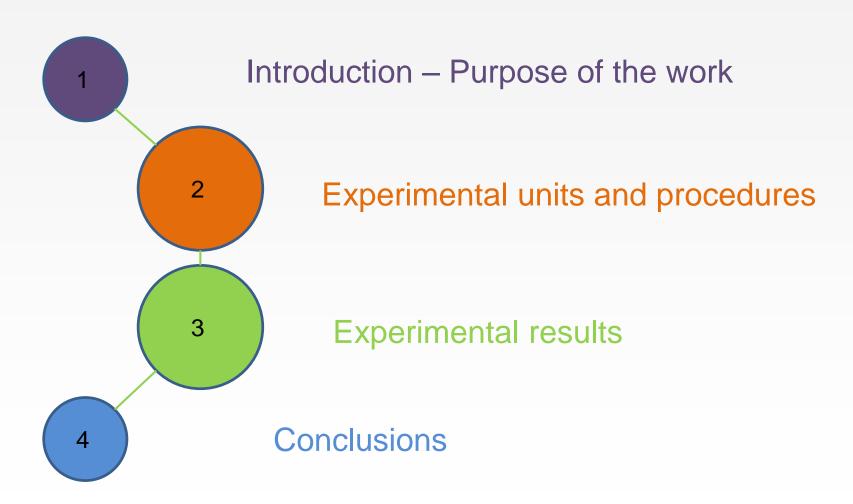
Dr. Topakas, Assoc. Professor

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## **Presentation Layout**

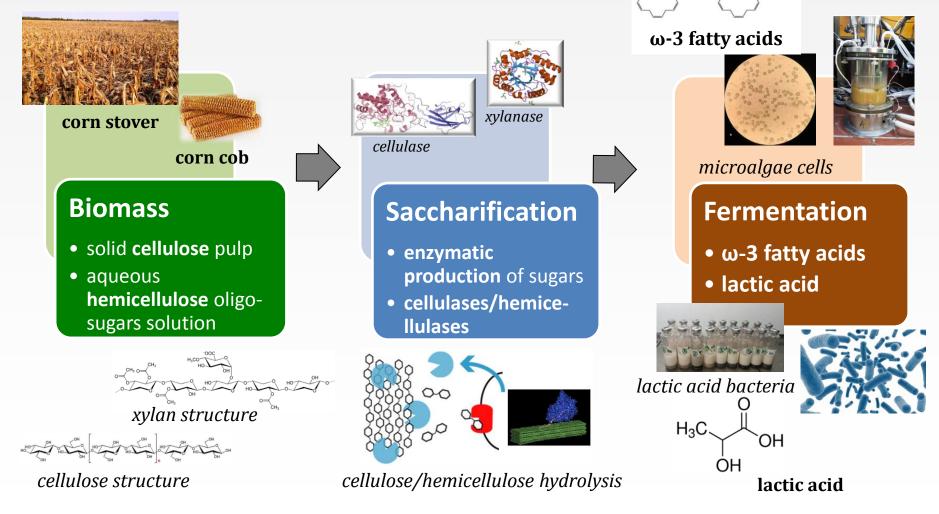


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## NoWasteBioTech Objectives



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#### **CPERI** Chemical Process and Energy Resources Centre for Research **Oxidative Organosolv**

## Acid Organosolv Delignification

lignin

lignin

OH

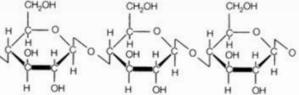
OMe

SO<sup>=</sup><sub>4</sub>

Vacuum distillation

Solid lignin

Delignification lignin OH 10 OMe lignin H<sub>2</sub>O/Acetone/Lignin one phase liquid Solid delignified pulp



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**Aqueous hemicellulose** 

byproducts solution

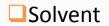


## Experimental results – Main parameters

### Biomass used was Lignocel HBS 150/500 which is a Beechwood sawdust

Extracts	A.I. Lignin	A.S. Lignin	Cellulose	Hemicellulose
3.7	21.7	2.5	47.6	21.2

#### **Main Parameters**



Pressure

#### Time

Temperature

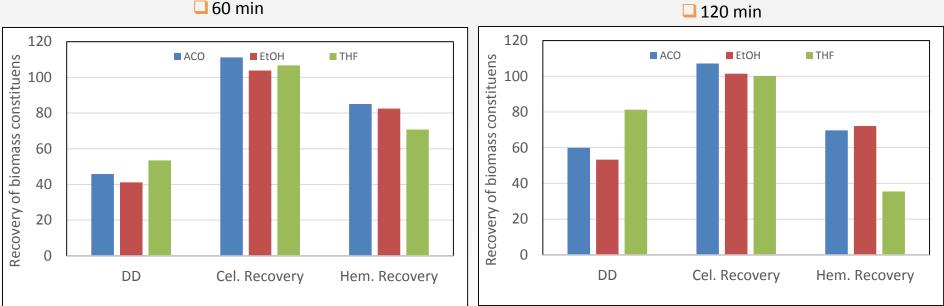
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## Solvent effect



#### **Main Parameters**

Solvent wt.%=50

LSR=10

- Solvent effect is significant, Acetone and THF very efficient, EtOH does not achieve high DD at low T
  - At higher reaction time, differences more pronounced

Cellulose recovery excellent in all cases (100%)

- □ 100% O<sub>2</sub> use
- □ T=150 °C
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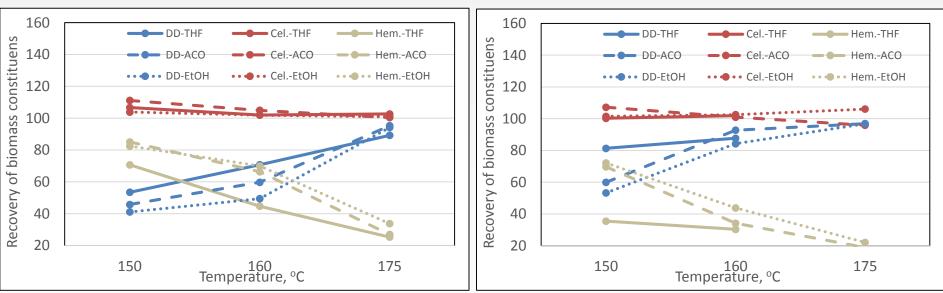
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🖵 t=60 min



### **Temperature effect**

🖵 t=120 min



#### **Main Parameters**

LSR=10

□ 100% O<sub>2</sub> use

🖵 t=60, 120 min

- Temperature has significant effect, especially at reaction time of 60 min
- 25 °C increase resulted in doubling of DD (~46  $\rightarrow$  95 %) at 60 min
- At 120 min even a 10 °C increase is enough to increase DD from 60 to 92%
- Cellulose recovery at 100% regardless of T

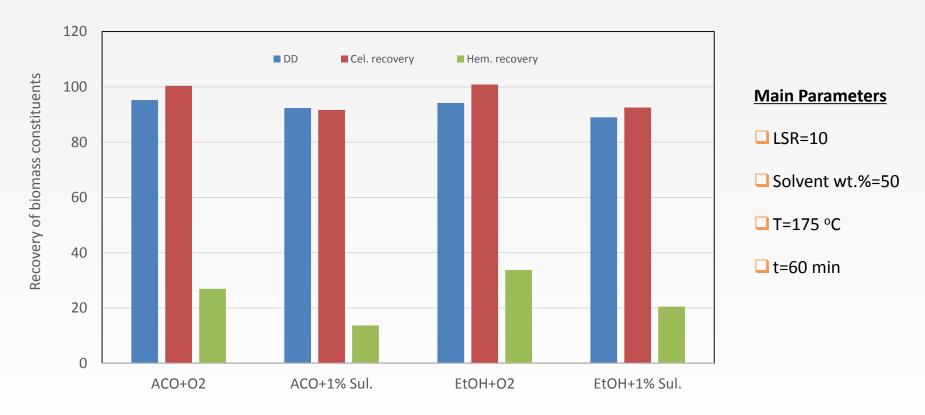
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## Acidic vs Oxidative Organosolv Delignification



- Use of O<sub>2</sub> instead of acids enhances delignification, up to 95% of lignin removed
- Cellulose recovery at 100% under O<sub>2</sub> delignification as opposed to ~92% under acidic delignification
- Hemicellulose recovery in pulp also increased with O<sub>2</sub> delignification due to less severe pretreatment

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## Fermentation to lactic acid

Simultaneous saccharification and fermentation (SSF) of *cellulose-rich biomass fractions* (>65% w/w cellulose) by Lactobacillus debruenckii sp. bulgaricus, following pretreatment

Lignocellulosic biomass (beechwood)		Biomass pretreatment		Hemicellulose %	Lactic acid (g/lt)	mg lactic acid/ g biomass	% theoretical yield
Mild-oxidative organosolv		H <sub>2</sub> O/ACO (50/50%), O <sub>2</sub> 8 bar, 160°C, 120min	66.77	18,36	77.11	756	79.60 ± 8.16
pretreatment	· •	H <sub>2</sub> O/ACO (50/50%), O <sub>2</sub> 16 bar, 160°C, 120min	76.63	13.32	61.07	678	67.67 ± 2.32
•		H <sub>2</sub> O/ACO (50/50%), O <sub>2</sub> 8 bar, 175°C, 120min	82.3	13.9	50.5	561	52.36 ± 1.01
Enzymatic hydrolysis with Cellic®CTec2 for sugar		H <sub>2</sub> O/ACO (50/50%), O <sub>2</sub> 16 bar, 175°C, 30min	79.74	15.69	64.56	717	67.43 ± 2.87
Dorduction		H <sub>2</sub> O/EtOH (50/50%), O <sub>2</sub> 16 bar, 160°C, 120min	72.96	16.03	52.5	583	58.72 ± 4.98
		H <sub>2</sub> O/EtOH (50/50%), O <sub>2</sub> 16 bar, 175°C, 60min	81.28	13.99	67.1	745	70.21 ± 0.91
	Í	H <sub>2</sub> O/THF (50/50%), O <sub>2</sub> 16 bar, 150°C, 120min	73.09	13.3	78.93	837	86.97 ± 6.7
		H <sub>2</sub> O/ THF (50/50%), O <sub>2</sub> 16 bar, 160°C, 120min	79.13	12.1	85.7	912	89.78 ± 10.1
		H <sub>2</sub> O/THF (50/50%), O <sub>2</sub> 16 bar, 160°C, 60min	68.99	15.55	72.96	785	83.29 ± 1.14
Fermentation for lactic acid production H <sub>3</sub> C U OH OH		H <sub>2</sub> O/ THF (50/50%), O <sub>2</sub> 16 bar, 175°C, 60min the most efficient in producing lactic a	85.28 acid with	10.82 high yields that	53.23 t reach up	591 to <b>0.91 g</b>	55.25 ± 0.42

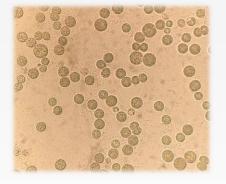
the most efficient in producing lactic acid with high yields that reach up to **0.91 g** lactic acid /g biomass after 168 h of fermentation, at 9% initial DM.



## Production of omega-3 fatty acids

Separate hydrolysis and fermentation (SHF) of *cellulose-rich biomass fractions* (>65% w/w cellulose) by *Crypthecodenium cohnii*, following pretreatment and enzymatic saccharification

Fatty acid composition
C14
C16
C18:0
C18:1
C22:0 (DHA)





#### The microalgae is capable of utilizing biomass sugars to grow and accumulate fatty acids.

Biomass pretreatment	Biomass (g/L)	Fatty acids (g/L)	Total fatty acids (% dry cell weight)	DHA (g/L)	DHA (% of total fatty acids)
THF/H <sub>2</sub> O (50/50), 160°C, 16 bar, 60 min	8.39 ± 0.09	$1.05 \pm 0.15$	17.6 ± 2.1	$0.47 \pm 0.12$	45.1 ± 2.8
EtOH/H <sub>2</sub> O (50/50), 160°C, 16 bar, 120 min	7.31 ± 0.29	0.65 ± 0.02	17.4 ± 3.2	0.36 ± 0.02	56.17 ± 1.9
ACO/H <sub>2</sub> O (50/50), 160°C, 8 bar, 120 min	9.03 ± 0.03	$1.41 \pm 0.08$	$14.4 \pm 0.9$	0.66 ± 0.04	46.74 ± 1.5
					×

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## Conclusions

### **Oxidative organosolv delignification**

- Use of O<sub>2</sub> instead of acids poses some advantages such as minimization of acidic wastes that require treatment, no need for corrosion resistant equipment.
- Oxidative delignification was very efficient at removing lignin (>95% DD).
- Cellulose recovery in solid form at 100%.
- Parameters effect is intertwined. Overall increase in temperature, O<sub>2</sub> pressure, time results in higher DD.
- Different solvents (acetone, ethanol, THF) efficient under different conditions.
- THF was very efficient at low T (150 °C), acetone was more efficient as T increased while ethanol needed higher T to perform well.
- Produced pulps successfully fed to microalgae and LA bacteria producing FA and LA.
- 0.91 g lactic acid/g biomass, >80g/lt lactic acid concentration, efficient production.
- >50% DHA in fatty acids produced by microalgae.



# Thank you for your attention! <u>kkalogia@cperi.certh.gr</u> http://nowastebiotech.cperi.certh.gr/





This project has received funding from the Hellenic Foundation for Research and Innovation (HFRI) and the General Secretariat for Research and Technology GSRT), under grant agreement No 1085

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