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Celabor: Walloon technological pilot platform for micro-algae refining and downstream processing

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Interreg France-Wallonie-Vlaanderen

GoToS3 ALPO

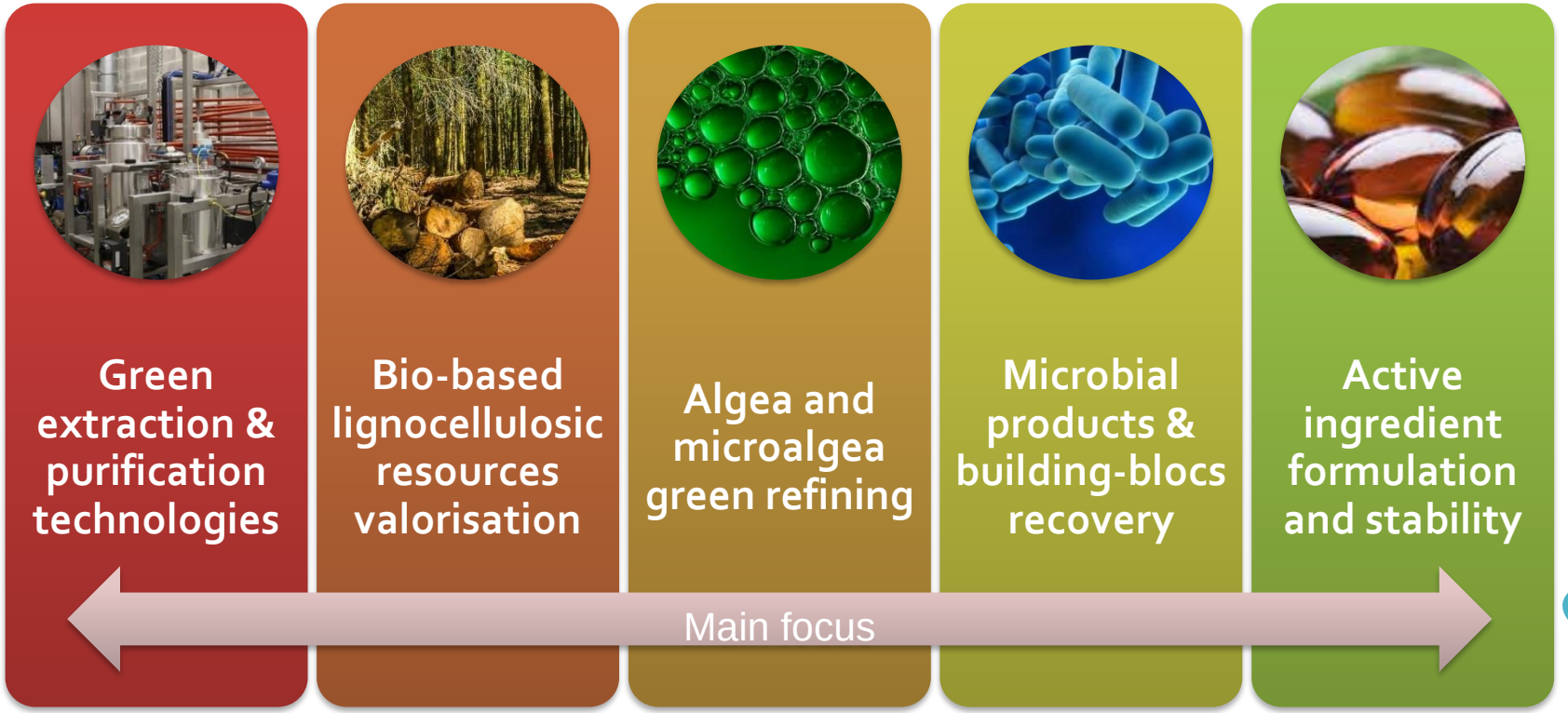
Workshop – Summer school 13th & 14th September 2018 – University of Mons
« Nouveaux matériaux polymères issus de la biomasse micro-algale »

CELABOR scrl. is a Belgian scientific and technical center based in the Petit-Rechain industrial park near Verviers.

CELABOR is accredited ISO 17025 by BELAC**,** CELABOR is offering scientific and technical support to companies involved in all sectors of **circular-economy** and **bioeconomy** including agrifood, green processes, packaging, textile and environment.

Five departments in the heart of the "Bioeconomy" sectors

"Food Technologies – Extraction" department



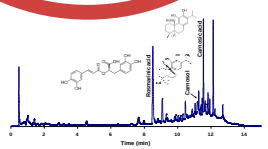
What we can do?

Pre-treatment and extraction of raw material

Fractionation and Purification

Post-treatment, concentration, drying

Characterization and quantification



Qualification and stability study

Technological pilot platform (350 m²)

Technological platform unique in Wallonia
ATEX zone

Two pilot plants **Supercritical Fluid Extractors SFE-CO2** (2x 6L/batch)

Pilot-scale **Subcritical Water Extraction** (SWE) (6L/batch)

Conventional **solvent extraction** (25L and 400L)

Pilot-scale **Pulsed Electric Field** (Solid: 0,5 kg/batch; liquid: 350L/batch)

Ultrasounds & Microwave Assisted Extraction (25L UAE/ 3L MAE)

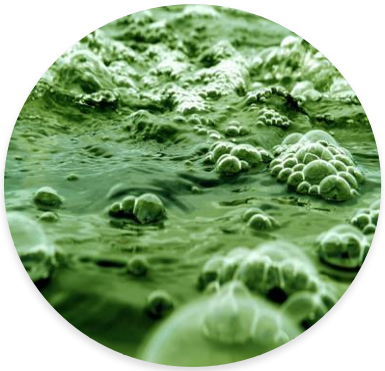
Lab and pilot-scale **membrane separation** (Ultra-filtration, Nano-filtration

Pilot-scale post-treatment equipment (Freeze-dryer, Spray-dryer, Evaporator, Centrifuge)

Purification platform (CPC, MPLC, Prep-HPLC)

Advanced **analytical lab** (UPLC-MS, GC-MS, ICP-MS, HPLC-DAD-ELSD

Main operations in microalgae refining & downstream processing



Pretreatment

Microalgal cell disruption step for cell permeabilization or break down. Techniques depends on the nature of the harvested microalgae: wet or dry

Extraction

Recovery of crude extracts using conventional and/or pressurized technologies depending on the nature of the microalgae and the application .

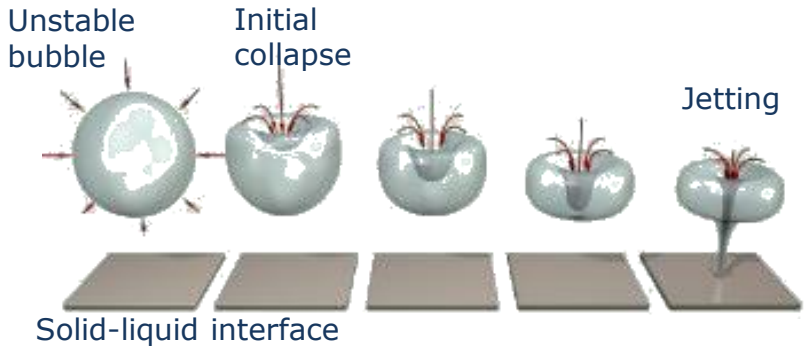
Purification

Fractionation and isolation of high added value compounds and/or family of compounds from the crude extract, *e.g.,* carotenoids, PUFA, proteins, EPS

Pretreatment Operations

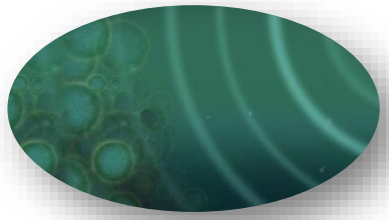
- Ultrasounds (US)
- High Pressure Homogenisation (HPH)
- Pulsed Electric Field (PEF)

Ultrasounds (US) for microalgal cell disruption



Ultrasound is a mechanical acoustic wave with the frequency range from roughly 10kHz to 20MHz.

- Cavitation is the main key for ultrasound intensification.
- The key to efficient application of ultrasound is control and selection of the energy intensity and population of active cavitation.
- The benefits of ultrasound for algal cell disruption is use of less energy consumption in comparison with high mechanical forces.
- Ultrasonic devices can be scaled up and operated on a continuous basis.



Scaling-up of microalgal cells disruption by ultrasounds technology at Celabor premises

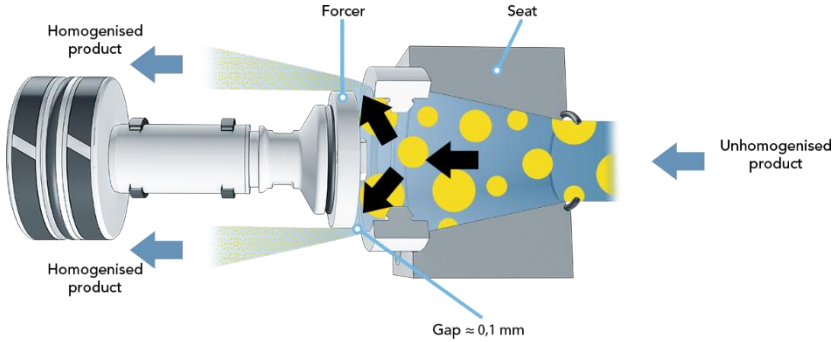
Lab-scale US device for batch operations with low frequency, transducers are fitted to the wall — **2 L**

Semi-pilot US for batch operations with mono-frequency **25 kHz**, transducers fitted to the wall — **25 L**

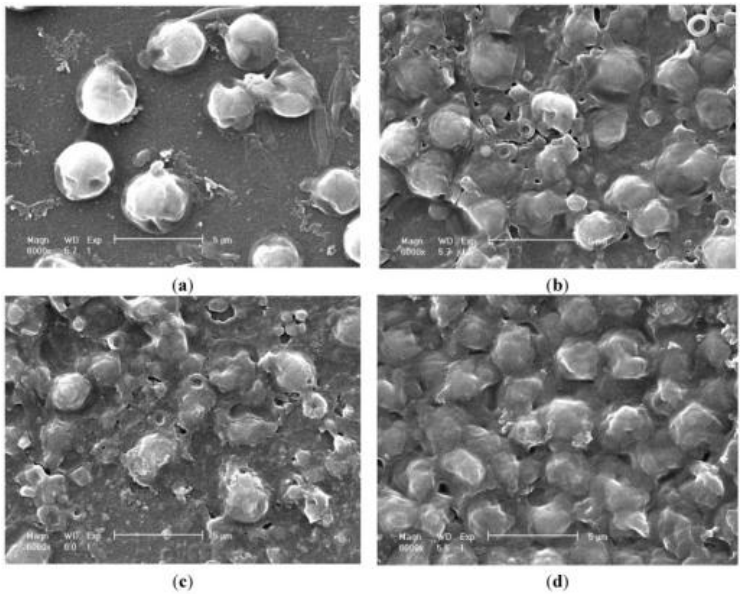
Semi-pilot US device for batch operations with low and high-frequencies, movable US probes — **50 L**

Pre-industrial new design US device, recirculation mode, continuous, multi-frequencies (Under development) — **400 L**

High Pressure Homogenisation (HPH) for microalgal cells disruption



*Olmstead ILD et al. (**2013**) Bioresource Technol. 148:615-619.*



Scanning electron microscopy (SEM) images of algae cell before and after disruption (6000×). (a) SEM image of algae cells of Neochloris oleoabundans; (b) SEM image of cells disruption with ultrasonic wave; (c) SEM image of cells disruption with high-pressure homogenization; and (d) SEM image of cells disruption with enzymatic hydrolysis.

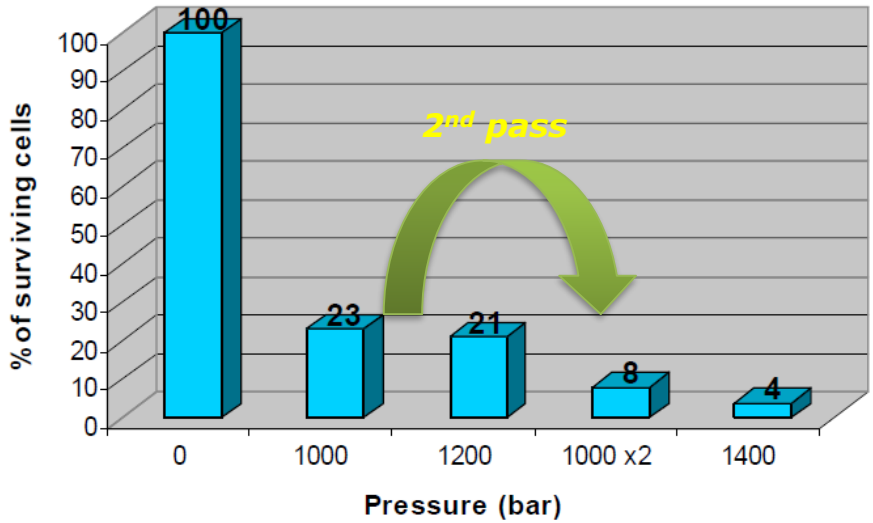
*Wang D, Li Y, Hu X, Su W, Zhong M (**2015**)- Int J Mol Sci*

Microalgal cells disruption by High Pressure Homogenisation (HPH) technology at Celabor premises

Panda Plus 2000 (GEA)
(9 L/h), Ex. Efficiency: More than 80% of disrupted cells (Chlorella sp.)

Mechanical rupture by high pressure homogenisation effective and efficient at high solids (20-25%)

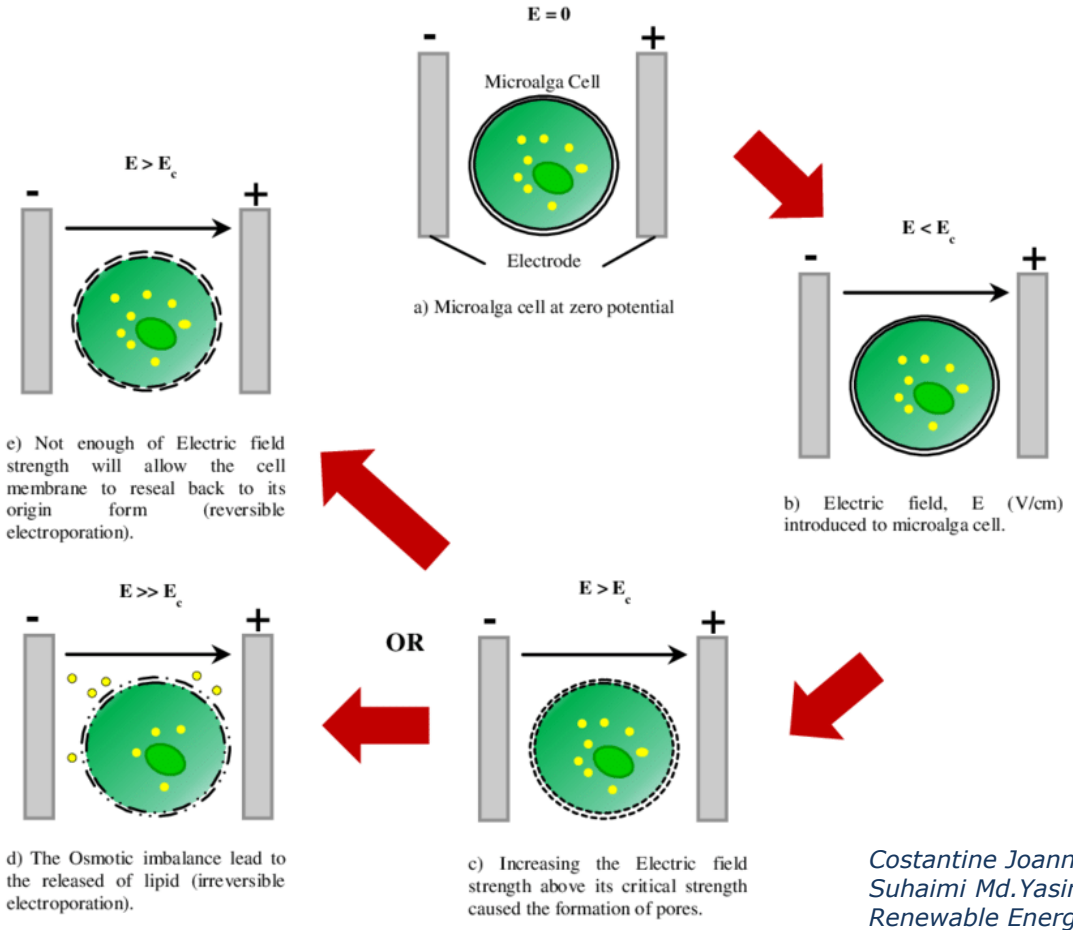
Chlorella high pressure disruption



Sample	Pressure (bar)	N° of cells	Rupture (%)
1	Not treated	53	Reference
2	1000	12	77 %
3	1200	11	79 %
4	1400	2	96 %

Efficiency of Chlorella high pressure disruption in function of pressure

Pulsed Electric Field (PEF): Eco-efficient tool for microalgal biomass pretreatment



Costantine Joannes, Coswald Stephen Sipaut, Jedol Dayou, Suhaimi Md.Yasir, Rachel Fran Mansa; International Journal of Renewable Energy Research-IJRER, ***(2015),*** *Vol 5, No 2*

Pulsed Electric Field (PEF): Eco-efficient tool for microalgal biomass pretreatment

20 kV, 12 kW (PEF) pilot plant (~ 300 L/h) available at Celabor

Improving quality with Pulsed Electric Field (PEF) Technologies

- PEF processing is an efficient **non-thermal pretreatment** technique
- Short, high voltage pulses, **cold extraction**
- Pulses induce poration of plant, animal and microbial cells, leading to cell disintegration and microbial inactivation
- Solid, semi-liquid and liquid materials can be treated

Tests on pilot plant equipment (350 L/h)

Innovative PEF products and applications for customers

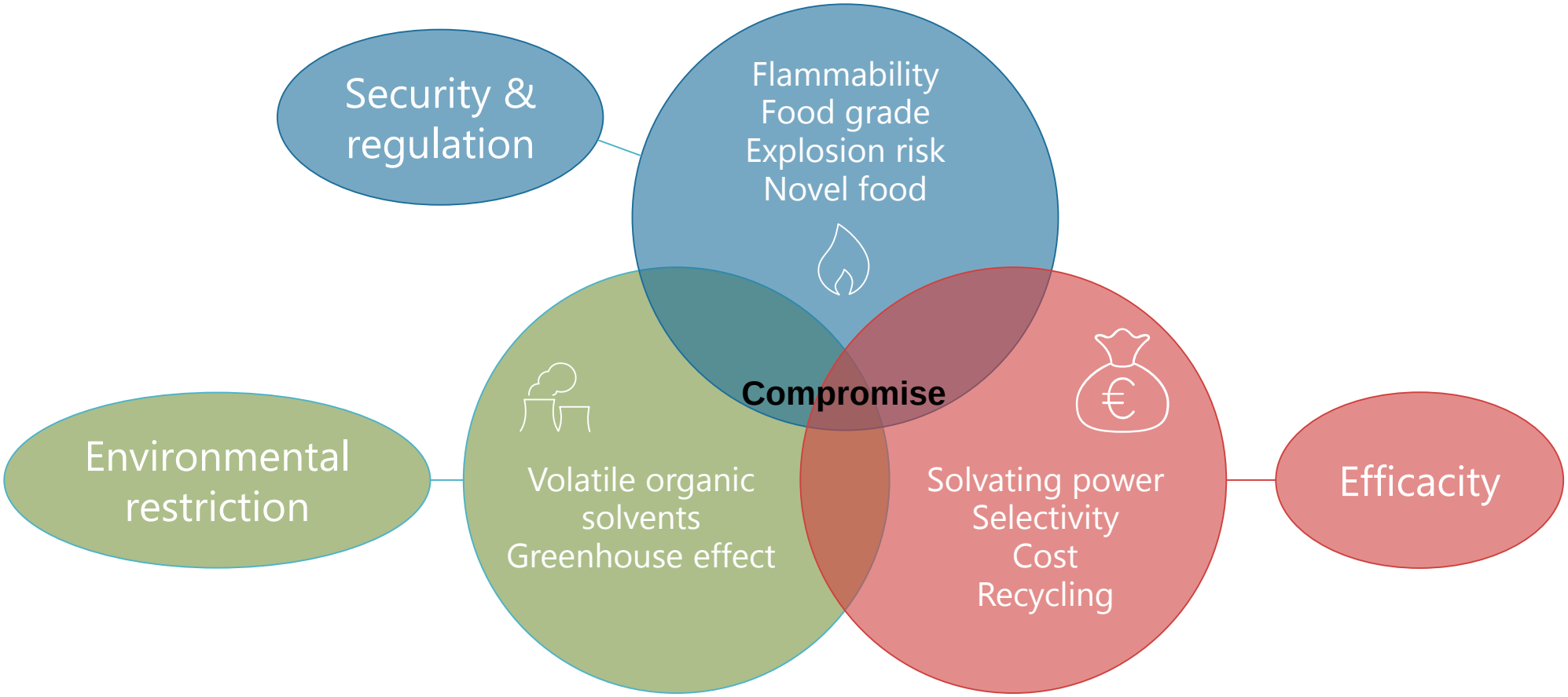
New applications development for cosmetics and other non-food sectors

Extraction

- Conventional solvent extraction
- Supercritical Fluid Extraction (SFE-CO2)
- Subcritical Water Extraction (SWE)

Conventional solvent extraction

Technical, sustainability and economical considerations



Microalgae extracts for cosmetic & food: limited options in terms of solvents

Scaling-up of microalgal actives recovery by solvent extraction technology in Celabor's ATEX zone

Laboratory-scale

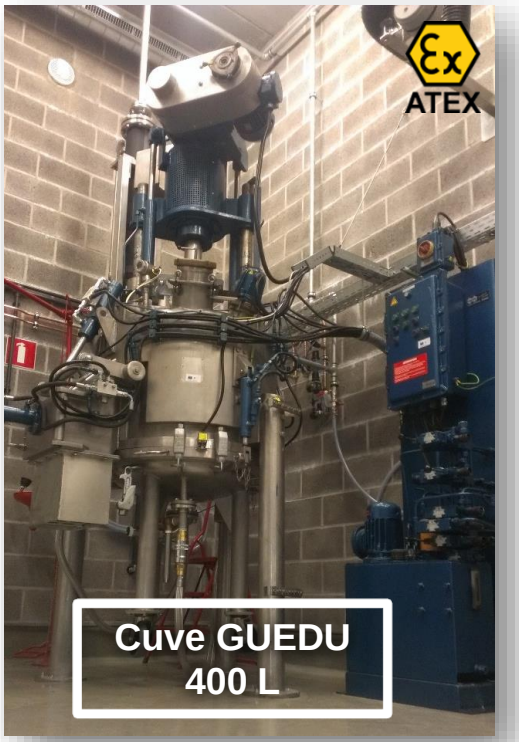
- From mg to some grams of material per batch
- Screening and optimizing extraction conditions

Semi-pilot scale

- Several hundreds of gram to some kilograms per batch
- Validation of pre-pilot production of target extract

Pilot-scale

- Several kilograms to several tenth of kilograms per batch
- Validation of pre-industrial production of target extracts






Pressurized "green" extraction processes

Supercritical CO2 - Subcritical water

Alternative solvents and technologies

Bio and agro solvents

- High cost, not always so efficient
- Bad HSE profile (health, security & environment)

Ionic-liquid solvents

- High cost
- Corrosive, non-volatile (distillation impossible)
- Possible eco- and cyto-toxicity in some cases

Fluorinated solvents

- Greenhouse effect
- High cost
- Recycling & purification difficult and costly
- Hazardous to handle

Pressurized solvents

- CO2-sc: industrial applications exist
- Subcritical water extraction: pilot units
- Cost

Supercritical fluids

Supercritical fluids

- Low viscosity and high diffusivity (gas)
- High density and high solvent power (liquid)
- Solvent polarity changes with pressure and temperature : fractionation

Carbon dioxide

Critical point for CO_2: 31°C – 73 bars

- Easy to reach
- Co-solvents use
- Industrial installations exist (TRL9)

Applicable

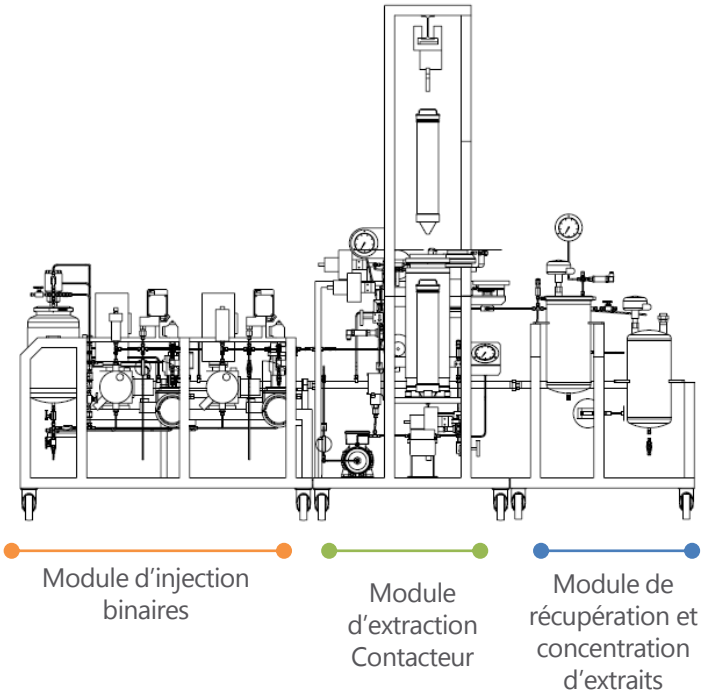
water

Critical point for H_2O: 374 °C – 221 bars

- Hard to reach
- Very corrosive
- Not suitable for bioactive extraction applications

Not applicable: Subcritical water

Supercritical CO_2: Celabor



Applications sc-CO_2

- Extraction of food ingredients (aromas, dyes, vitamin, specific lipids, …)
- Extraction of aroma from fermented and distilled beverages
- Deodorisation/decoloration of natural extracts
- Debacterization of beverages and fresh vegetables

Supercritical Fluid Extraction applied to microalgae

ELSEVIER

Bioresource Technology

Volume 269, December 2018, Pages 81-88

Selectively biorefining astaxanthin and triacylglycerol co-products from microalgae with supercritical carbon dioxide extraction

Thomas Alan Kwan [a], Sarah Elizabeth Kwan [a], Jordan Peccia [a], Julie Beth Zimmerman [a, b]

Journal of Food Engineering 116 (2013) 478–482

Contents lists available at SciVerse ScienceDirect

Journal of Food Engineering

journal homepage: www.elsevier.com/locate/jfoodeng

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Research note

Supercritical fluid extraction of carotenoids and chlorophylls *a*, *b* and *c*, from a wild strain of *Scenedesmus obliquus* for use in food processing

A. Catarina Guedes [a,b], Maria S. Gião [a], Ana A. Matias [c,d], Ana V.M. Nunes [c,d], Manuela E. Pintado [a], Catarina M.M. Duarte [c,d], F. Xavier Malcata [c,e,*]

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The Journal of Supercritical Fluids

Volume 79, July 2013, Pages 337-344

Influence of pretreatment on supercritical CO_2 extraction from *Nannochloropsis oculata*

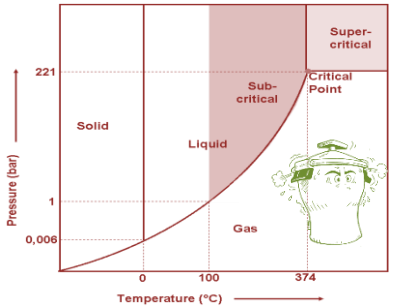
Christelle Crampon [a], Adil Mouahid [a], Sid-Ali Amine Toudji [a], Olivier Lépine [b], Elisabeth Badens [a]

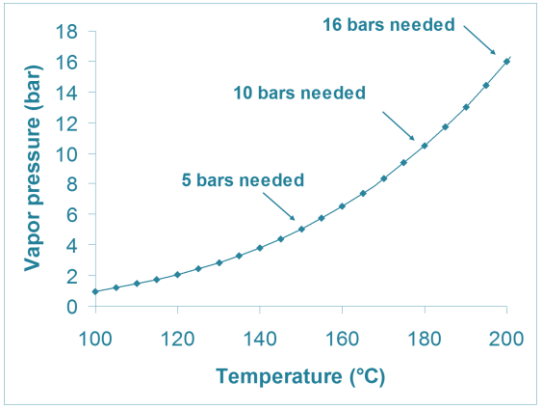
Rigid microalgae need high pressure (800 to 1000 bar) ➔ high cost & problem for industrialisation

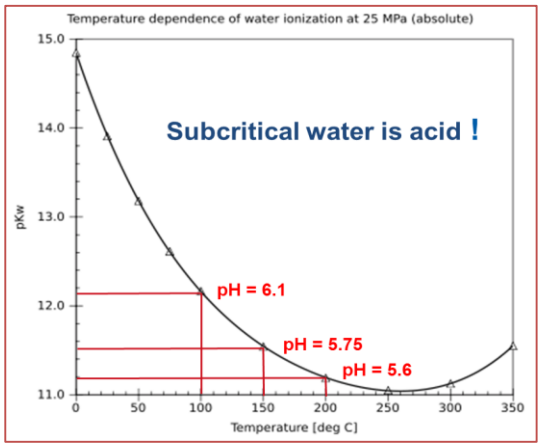
Low yield is obtained when extracting untreated (intact) microalgae (scenedesmus sp., Nannochlorepsis sp.) with highly rigid cell membrane) ➔ PEF, HPH, etc., before sc-CO2 – induce almost total recovery.

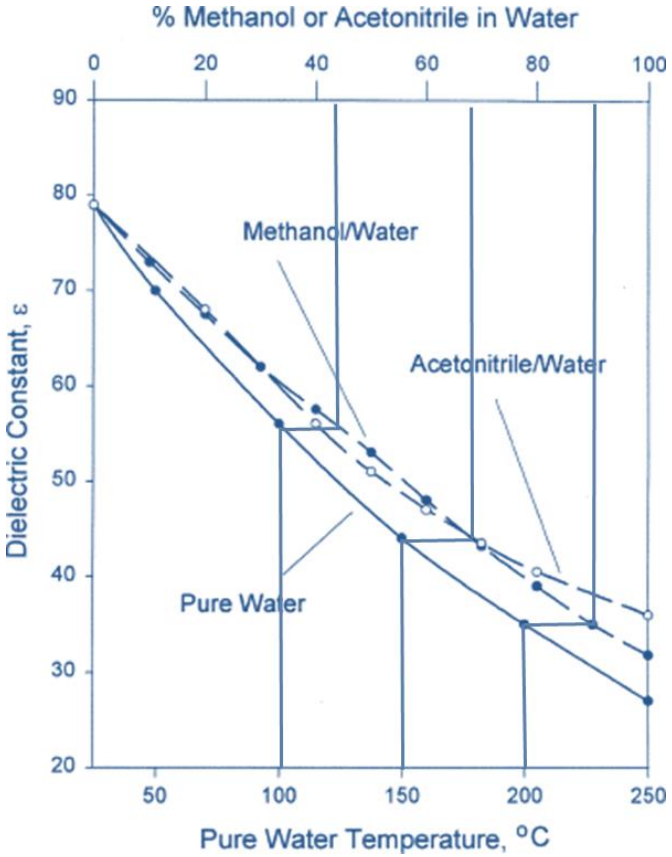
Subcritical Water Extraction?

Superheated water is liquid water under pressure at temperatures between the usual boiling point, 100°C and the critical temperature, 374°C. It is also known as "subcritical water" or "pressurized hot water."









Characteristics & advantages

- Faster diffusion and lower viscosity
- Non-flammable, non-toxic
- Cheaper (ethanol)
- Applications: Food and cosmetics
- Better solubility of less polar compounds (polyphenol, flavonoid, essential oil…)
- Reaction of hydrolysis (lignocellulosic material: hemicellulose)

Limitations

- Availability of industrial installations (under development)
- Degradation of themo-labile compounds

Scaling-up of SWE technology in Celabor's ATEX zone

Lab-scale developments

Pilot-scale developments and up-scaling
Working zone : 100°C – 200°C

Extraction of high added value compounds : polyphenols,…(no EtOH)
Hydrolysis of the lignocellulosic content : hemicellulose

Subcritical Water Extraction applied to microalgae

Journal of Pharmaceutical and Biomedical Analysis
Volume 51, Issue 2, 20 January 2010, Pages 456-463
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Subcritical water extraction and characterization of bioactive compounds from *Haematococcus pluvialis* microalga

I. Rodríguez-Meizoso [a], L. Jaime [b], S. Santoyo [b], F.J. Señoráns [b], A. Cifuentes [a], E. Ibáñez [a]

BioMed Research International
Volume 2016, Article ID 5816974, 10 pages
http://dx.doi.org/10.1155/2016/5816974

Research Article

Subcritical Water Technology for Enhanced Extraction of Biochemical Compounds from *Chlorella vulgaris*

S. A. Awaluddin,[1] Selvakumar Thiruvenkadam,[1] Shamsul Izhar,[1] Yoshida Hiroyuki,[1] Michael K. Danquah,[2] and Razif Harun[1]

[1]Department of Chemical and Environmental Engineering, Universiti Putra Malaysia, 43400 Serdang, Malaysia
[2]Department of
Malaysia

(12) **United States Patent**
Deng et al.

(10) **Patent No.: US 9,328,310 B1**
(45) **Date of Patent: May 3, 2016**

(54) **SUBCRITICAL WATER EXTRACTION OF LIPIDS FROM WET ALGAL BIOMASS**

(71) Applicant: **Arrowhead Center, Inc.**, Las Cruces, NM (US)

(72) Inventors: **Shuguang Deng**, Las Cruces, NM (US); **Harvind K. Reddy**, Las Cruces, NM (US); **Tanner Schaub**, Las Cruces, NM (US); **Francisco Omar Holguin**, Las Cruces, NM (US)

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Bioresource Technology
Volume 131, March 2013, Pages 413-419
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Hydrothermal liquefaction of *Spirulina* and *Nannochloropsis salina* under subcritical and supercritical water conditions

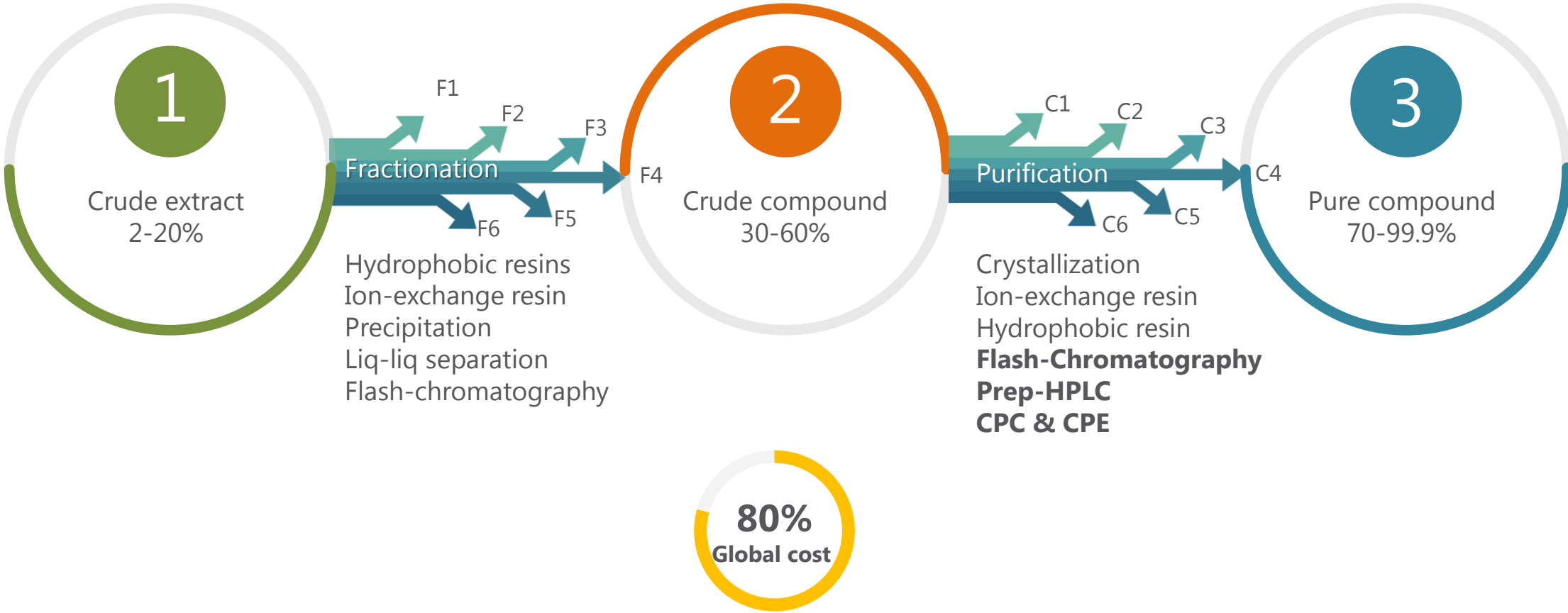
Saqib S. Toor [a], Harvind Reddy [b], Shuguang Deng [b], Jessica Hoffmann [a], Dorte Spangsmark [c], Linda B. Madsen [c], Jens Bo Holm-Nielsen [d], Lasse A. Rosendahl [a]

The successful use of water as a "green" solvent indicates that SCW extraction of lipids and bioactives from microalgae is an environmentally friendly alternative to traditional solvent-based extraction methods.

Fractionation - Purification

- Flash-Chromatography
- Prep-Liquid Chromatography
- Centrifugal Partition Chromatography

Resins & Chromatographic Techniques



80%
Global cost

Chromatographic Techniques - CELABOR

Dynamic Axial Compression (DAC) Prep-HPLC system

Fully automated CPC & CPE "GILSON"

Celabor - Preparative Chromatography lab

RESEARCH PROGRAM & COLLABORATIVE PROJECTS

The main mission of Celabor is to encourage technological innovation and the development of new products or processes through research and development. Celabor conducts private development and research on behalf of companies and also participates in research and development programs financed by Europe and the Regions.

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