AFTERLIFE

OCTOBER 15, 2019

WEBINAR- Advanced Filtration Technologies for the Recovery and Later conversion of relevant Fractions from wastewater

MARÍA LÓPEZ

idener
SCIENTIFIC COMPUTING

BBI JU

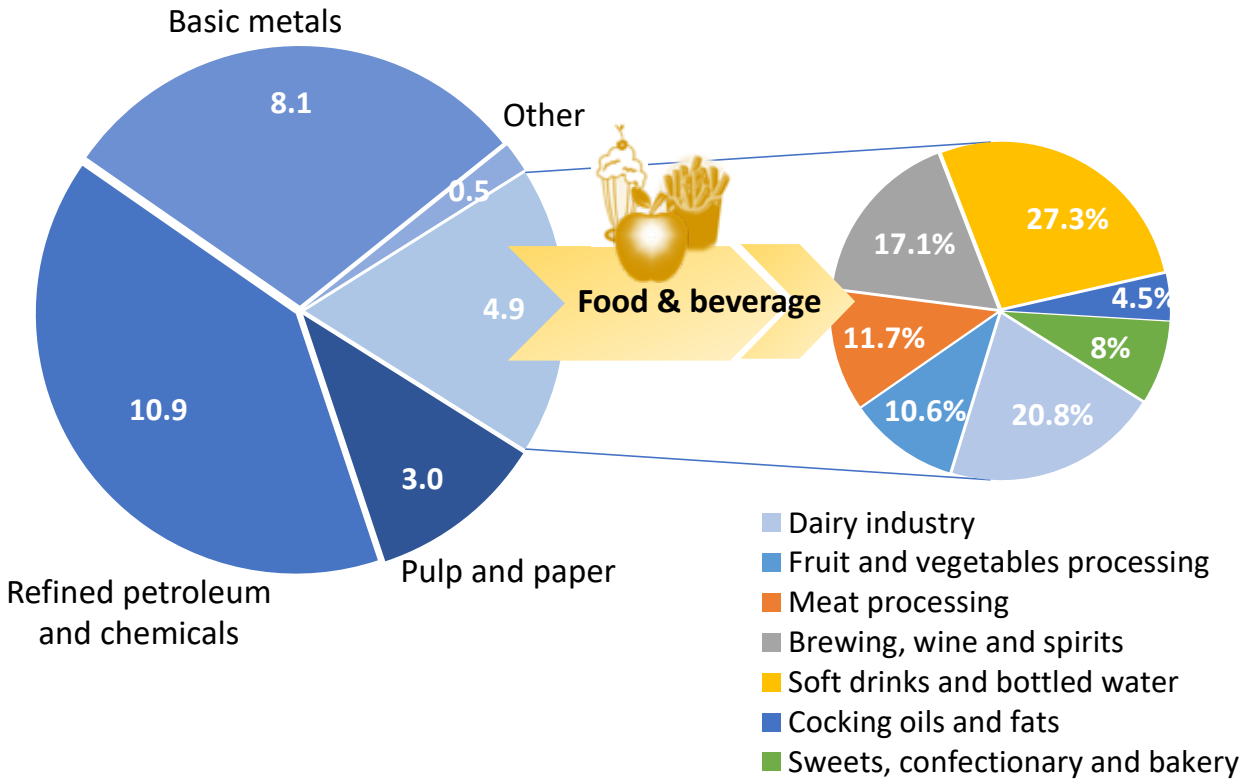
Bio-based Industries Consortium

Horizon 2020
European Union Funding
for Research & Innovation

AFTERLIFE has received funding from the Bio-Based Industries Joint Undertaking under the European Union's Horizon 2020 research and innovation program under grant agreement No. 745737 .

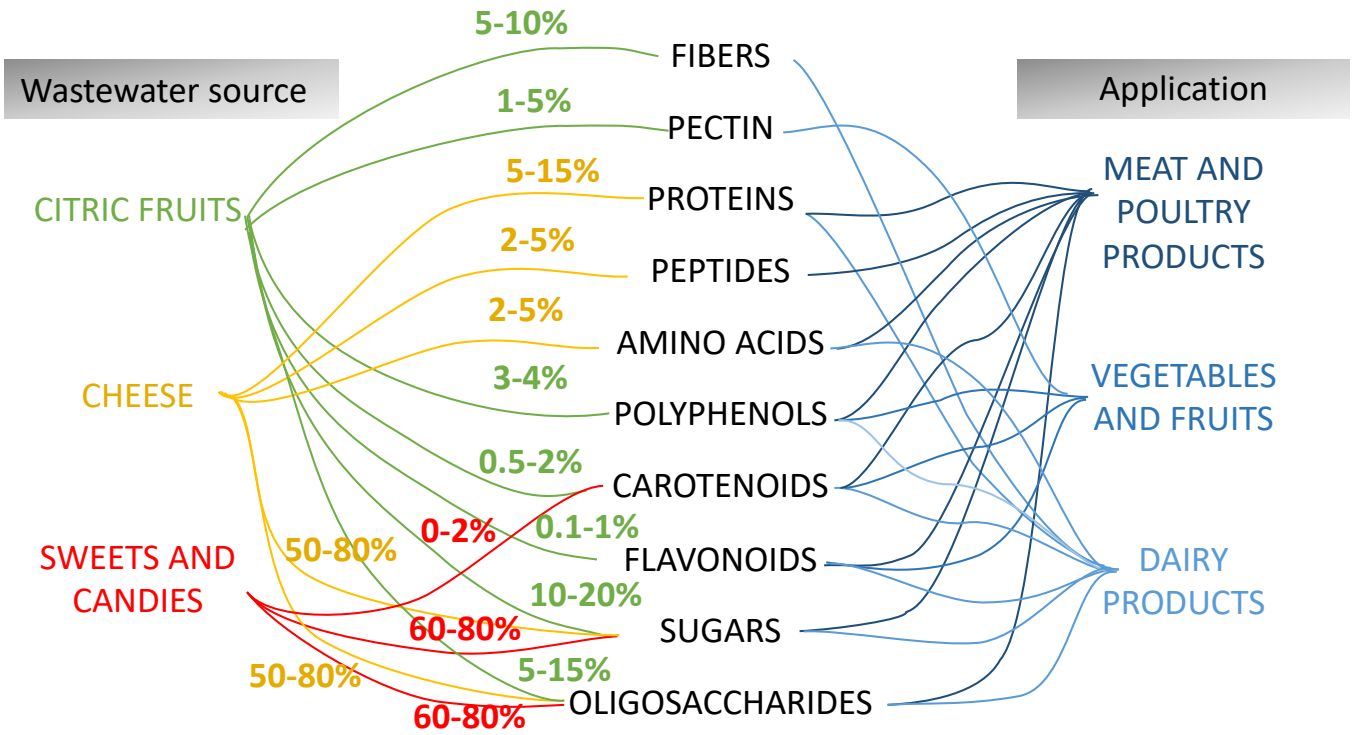
Welcome
and scope of the webinar

Wastewater production in European industries



Welcome
and scope of the webinar

Wastewater from food processing: a great source of bio products!!



Welcome
and scope of the webinar

Focus on extraction and concentration techniques that will lead to the valorization of wastewater

- Green techniques
- Cost-effective
- Flexible

Value-added products

Reusable water

Application of extraction techniques in wastewater valorisation: AFTERLIFE project

- The AFTERLIFE project proposes a flexible, cost- and resource-efficient process for valorizing wastewater
- It will represent an advance on existing approaches to wastewater treatment
- It will separate out the different components of value using a series of membrane filtration units
- These will then be treated to obtain high-pure extracts and metabolites or, alternatively, to be converted into value-added biopolymers
- In addition to the value extracted from the solids, the remaining outflow of the water will be ready for re-use

Project Consortium

4
Duration (Years)

3.890.000
Max. grant amount

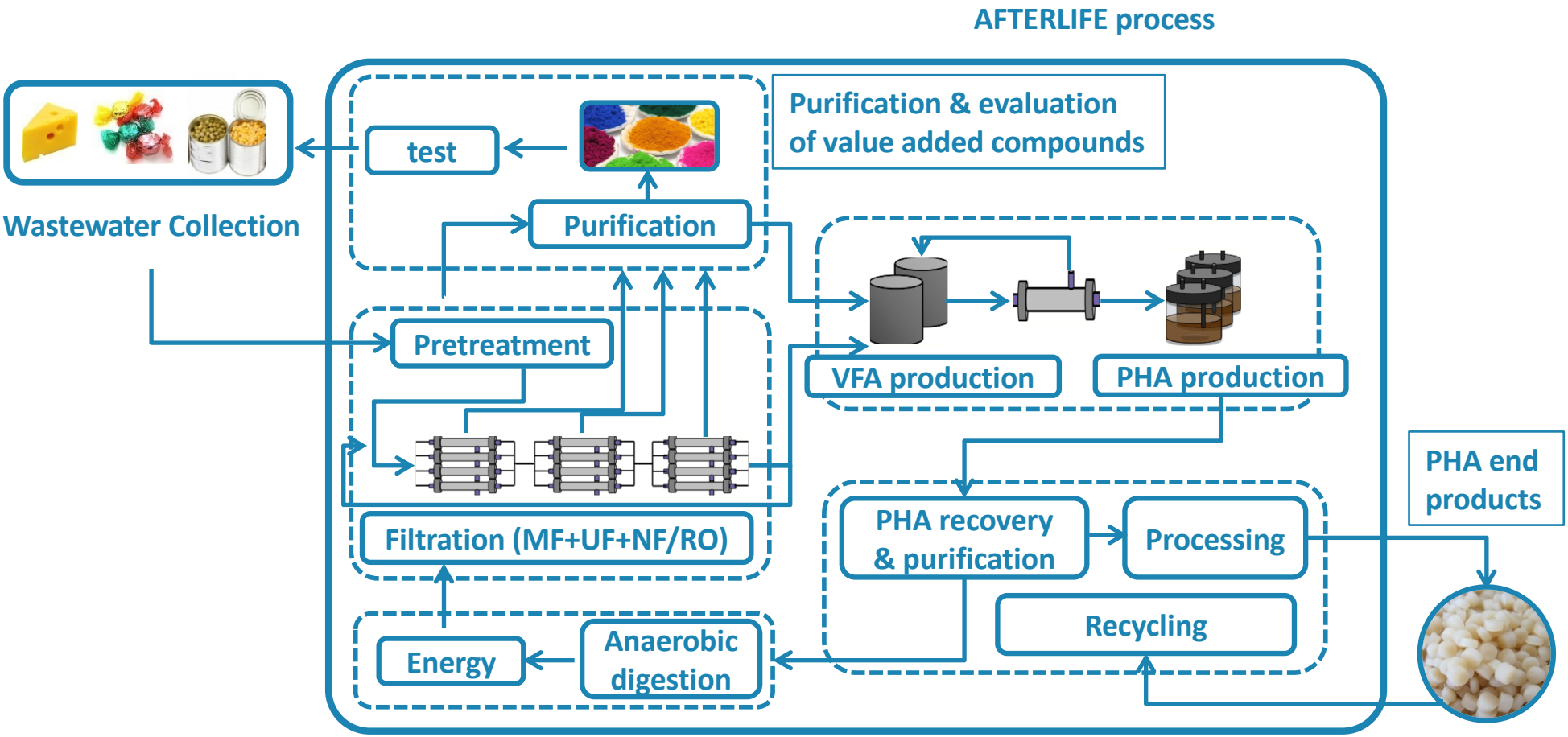
14
Partners

7
Countries

5 RTD &
Non-profit org.

9 SMEs

The process

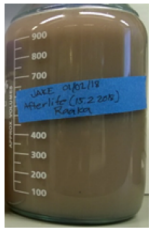

AFTERLIFE process
Wastewater Collection
test
Purification & evaluation of value added compounds
Purification
Pretreatment
VFA production
PHA production
Filtration (MF+UF+NF/RO)
PHA recovery & purification
Processing
PHA end products
Recycling
Energy
Anaerobic digestion

AFTERLIFE project: wastewater

Wastewater Collection

Heritage-W

Jake-WW



Citromil-JL

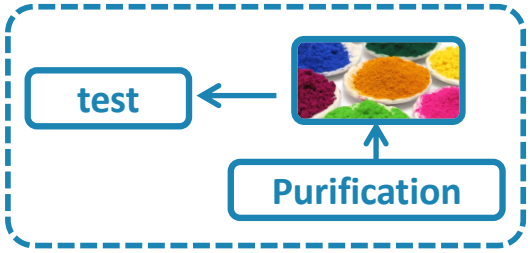
Citromil-EO

- **High concentrations of SS**
- Whey can be studied as a **raw material of fat, protein and lactose**

- **High concentration of SS**
- **Very high sugar content**
- Low fat and protein concentrations

- Higher concentrations of compounds in Cit-EO than Cit-JL
- **Notable SS/pulp** concentration
- Some sugars, low fat and low protein concentrations
- **High concentrations of compounds of interest** in Citromil-EO, **such as flavonoids and limonoids, and relevant quantities of essential oils**

Extraction and purification



- Essential oils extraction with (microwave) distillation
- Use of enzymatic hydrolysis and pH modification for the extraction and fractionation of the compounds of interest (flavonoids, limonoids)
- Use of commercial resins for the refining (purity at least 4 times higher than the initial extract)
- Residue valorisation
- Tests for food applications

Webinar Speakers

Mr. Thibaut Derycke
Team Leader Bioprocessing – BBEU

FILTRATION, SOLVENT EXTRACTION AND STEAM EXTRACTION, WITH AN EYE ON THE BIG NUMBERS

Dr. Javier Ceras
Technical Investigator – Lurederra

RECOVERY OF NATURAL COMPOUNDS OF INTEREST FROM AGRIFOOD WASTES

Stakeholder Workshop

Save the dates for the AFTERLIFE stakeholder workshop at the 13th international conference on bio-based materials 12-13th May 2020

AFTERLIFE

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FILTRATION, SOLVENT EXTRACTION AND STEAM EXTRACTION, WITH AN EYE ON THE BIG NUMBERS

Thibaut Derycke
thibaut.derycke@bbeu.org

Bio Base Europe Pilot Plant

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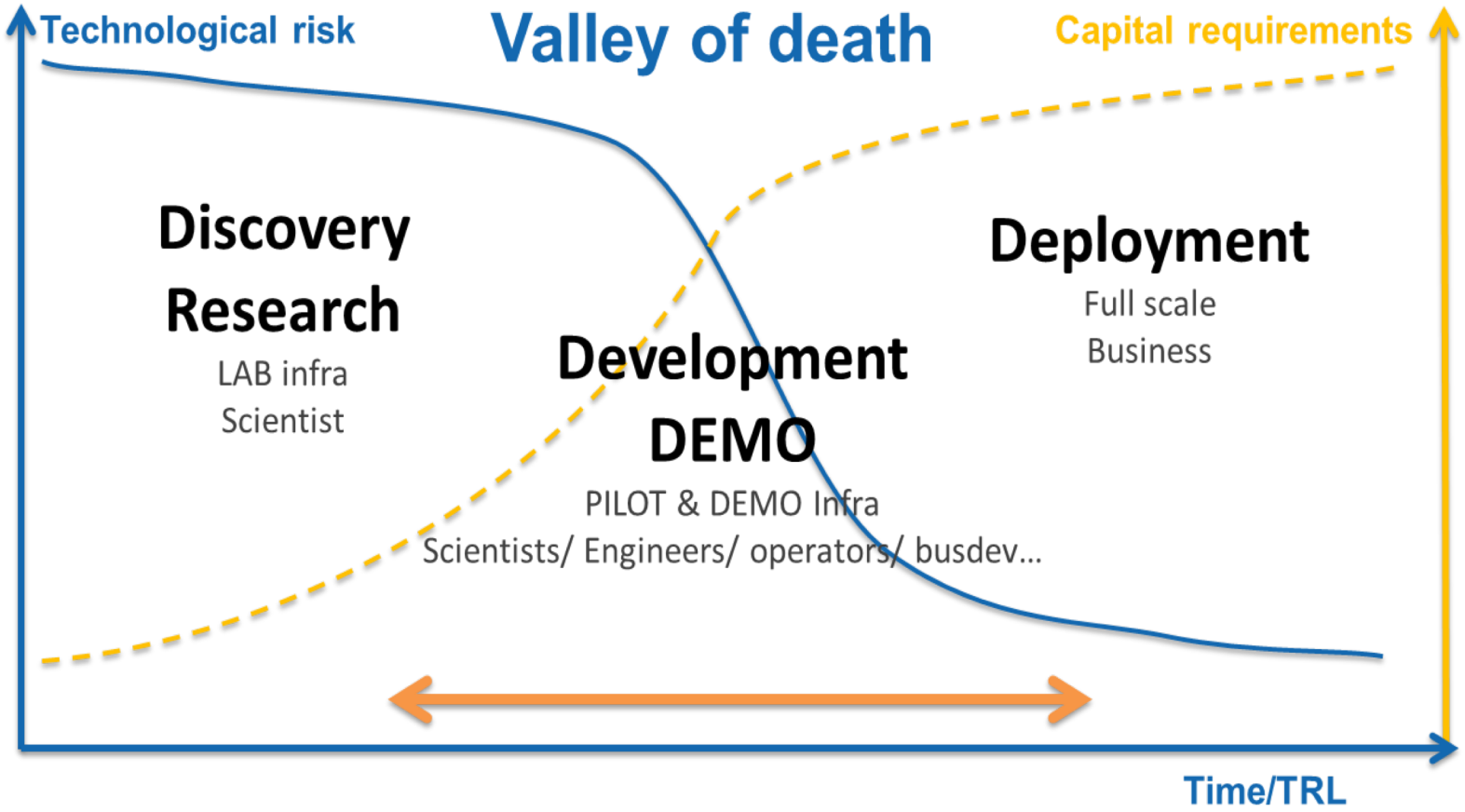
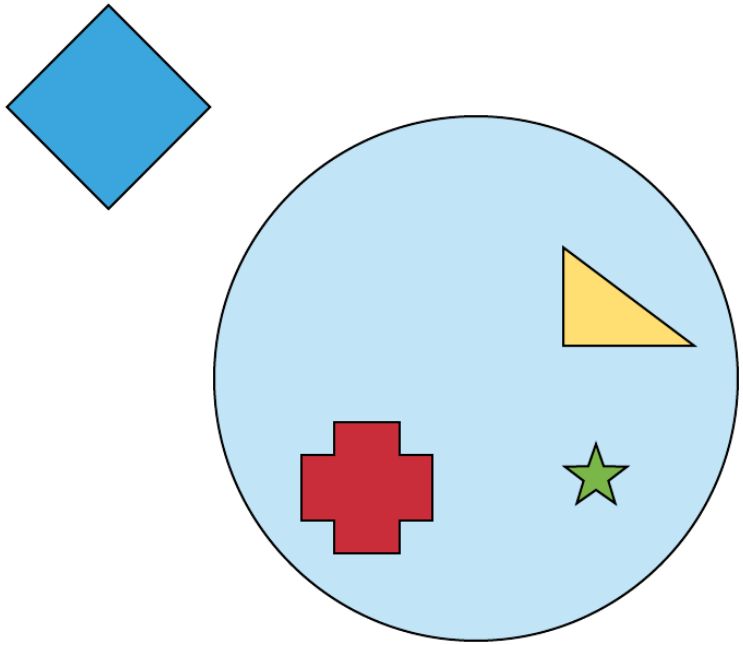
Valley of death



Define your stream

- Components of interest:
  - Size
  - Charge, IEP
- How many components are there to purify?
- How many components you do not want to purify?
- Purity requirements?



Multiple techniques

Size exclusion

- Dead end filtration
- Cross flow filtration
- *Resin technology*

Affinity

- Solvent extraction
- Steam extraction
- (Steam) distillation
- *scCO2 extraction*
- *Ionic liquids*
- *Affinity chromatography*

Density

- *Centrifugation*
- *Decantation*
- *Flotation*
- *Flocculation*

Iso electric point (I.E.P)

- *electrophoresis*

Multiple techniques

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Size exclusion

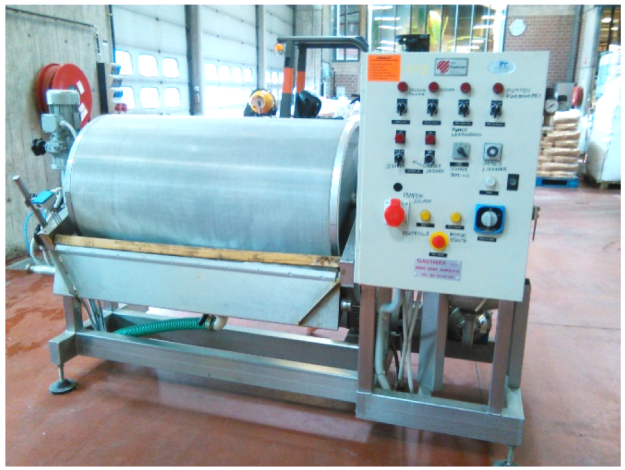
Dead end filtration

Fibrous materials

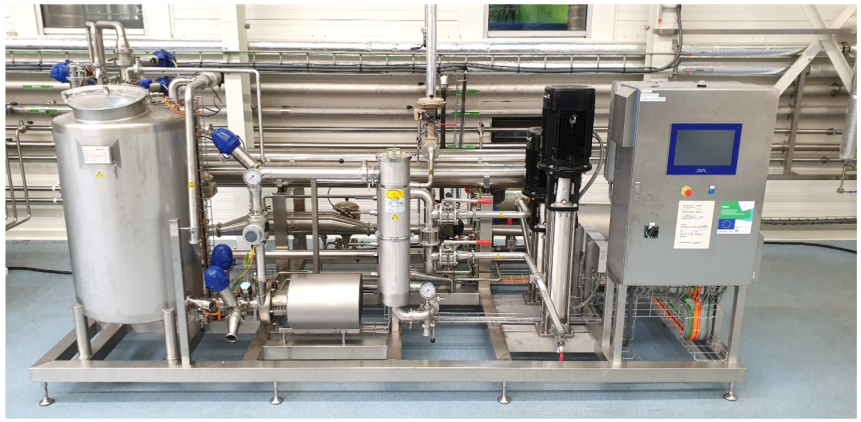
Low retentate/permeate ratio

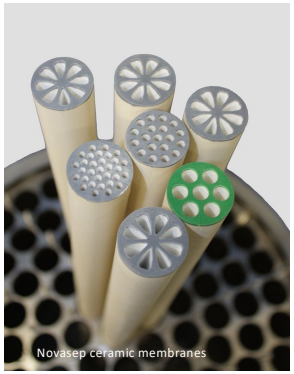
+ cheap membranes
+ straightforward
+ dry retentate

- Fouling
- Low throughput
- Not continuous
- Labour intensive
- Cleaning
- Can become expensive



Size exclusion





Cross flow filtration

Fibrous materials

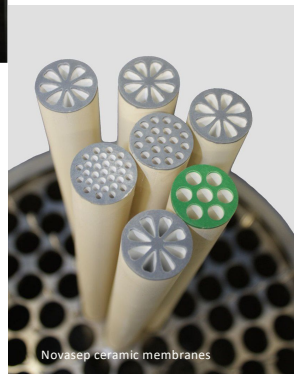
High rententate/permeate ratio

\+ High throughput
\+ Space-time optimal
\+ Continuous
\+ Less prone to fouling
\+ Cleanability

\- More expensive
\- Pre-purification might be required
\- Dead volume

Size exclusion


Alfa laval SW membranes


Novasep ceramic membranes

Types

Spiral wound

- Highest surface/space ratio
- Requires clean feed
  => strainers
- High TMP possible 40-50 bar
  (think RO)

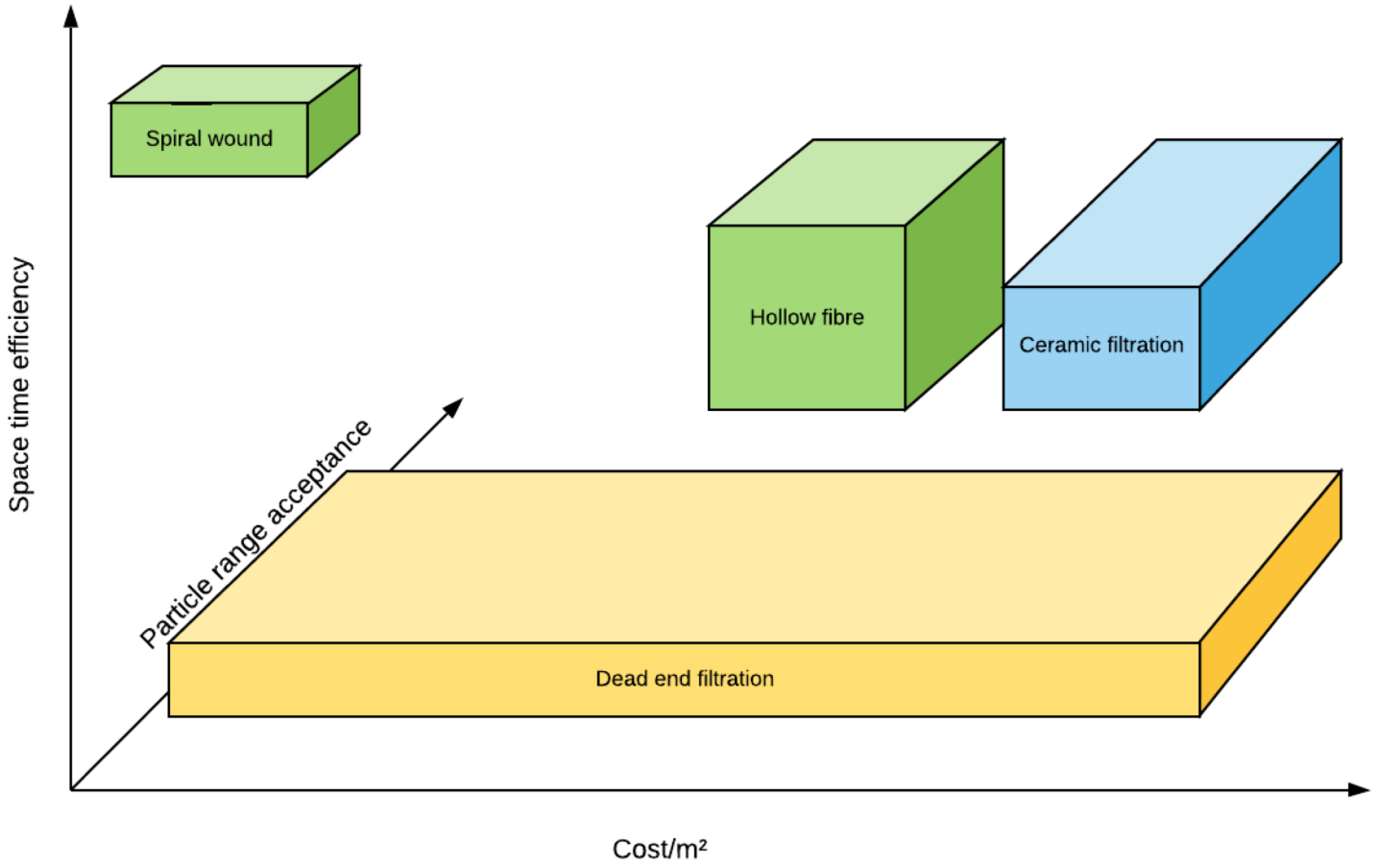
Hollow fibre

- Lower surface/space ratio
- Can deal with less clean feeds
- Medium TMP (+-3 bar)

Ceramic

- Lower surface/space ratio
- Most robust cross flow type
- Medium TMP (3-5 bar)

Cost drivers

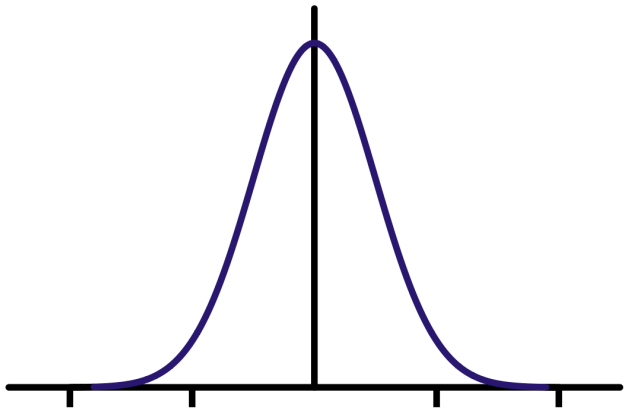


Important parameters

- Cut-off
- Flux: 10 L/m²/h/bar
- Material choice:
  - PES, PA, CA, PS, PVDF, PTFE, TFC, PP, TFC PA,…
  - Compatibility with product
- Fouling of membranes: L/m²

⇒ Filter aids

⇒ Coagulating agents



Multiple techniques

Size exclusion

- Dead end filtration
- Cross flow filtration
- *Resin technology*

Affinity

- Solvent extraction
- Steam extraction
- Vacuum extraction
- *scCO2 extraction*
- *Ionic liquids*
- *Affinity chromatography*

Density

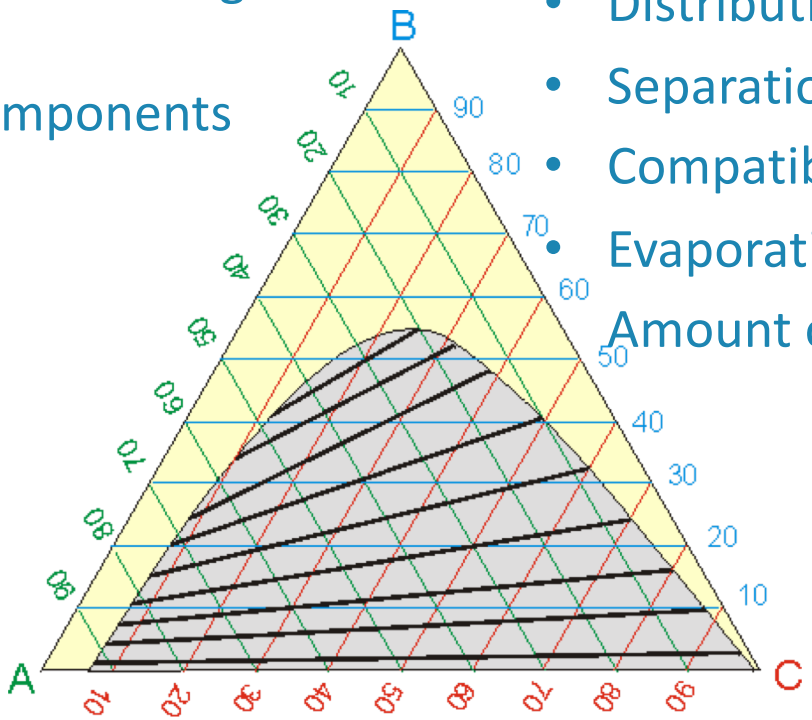
- *Centrifugation*
- *Decantation*
- *Flotation*
- *Flocculation*

Iso electric point (I.E.P)

- *electrophoresis*

Solvent extraction

+ continuous
+ multi stage possible
+ Low energy input
- EX
- High quantities of solvent might be required
- Extraction other components
- Cost of solvent



Parameters:

- Feed rate
- pH aquous phase and pKa components
- Distribution ratio
- Separation extract/raffinate
- Compatibility with equipment
- Evaporation energy solvent
- Amount of stages required

Steam distillation

Uses:

- Less thermostable components
- High boiling points matrices
- F.e. essential oils, aromatics

Pluses

+ straightforward

+ High heat transfer

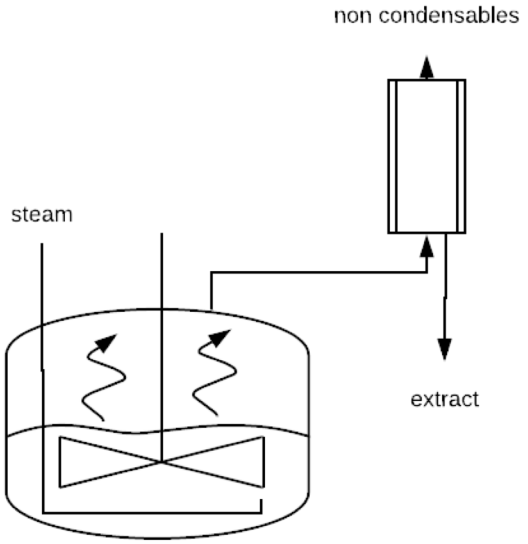
+ More energy efficient compared to hydrodistillation

Downsides

- Often replaced by vacuum distillation, as energy cost is still relatively high
- separation of water/ organic phase required

Parameters

- Steam temperature
- Pressure
- Affinity
- DM% of material



(Vacuum) Distillation

Uses:

Separation components with different vapour points

Vacuum distillation:

- Distillation at reduced pressure
- Increased differences of vapour pressures
- Lower CAPEX
- Slightly higher OPEX
- More efficient (fewer stages)

Parameters

- Feed(Q, T, X)
- Composition distillate
- Composition raffinate
- Design distillation column
  - Reflux/reboil ratio
  - Positioning feed
  - Amount of trays
  - Packing material

THANK YOU!

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RECOVERY OF NATURAL COMPOUNDS OF INTEREST FROM AGRIFOOD WASTES

Dr. Javier Ceras
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Lurederra centro tecnológico

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Agri-Food Industry Wastes

- What

  Wastewaters

  By-products: considered wastes in most cases

- Why

  Huge amount of material

  Important source of high added value compounds

  Existing technologies are able to recover these valuables

- But…

  Lab scale

  Marketable produts are still rare

Extraction Technologies

Green Extraction

(Chemat 2012)

processes which will reduce energy consumption,

allows use of alternative solvents and renewable natural products,

and ensure a safe and high quality extract/product

Identified solutions

improving and optimization of existing processes

using non-dedicated equipment

innovation in processes and procedures but also in discovering alternative solvents

Extraction Technologies

- What's the objective?

Different chemistry of compounds of interest

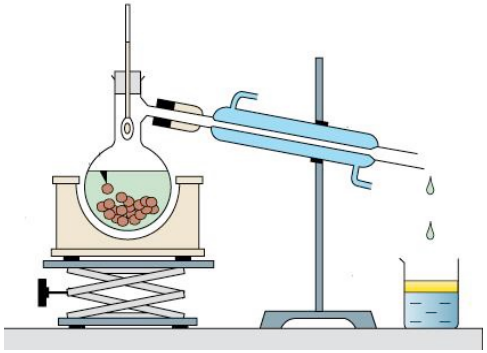
Co-extraction of different compounds

- Definition of target compounds

AFTERLIFE Citromill Essential Oil Line Wastewaters:

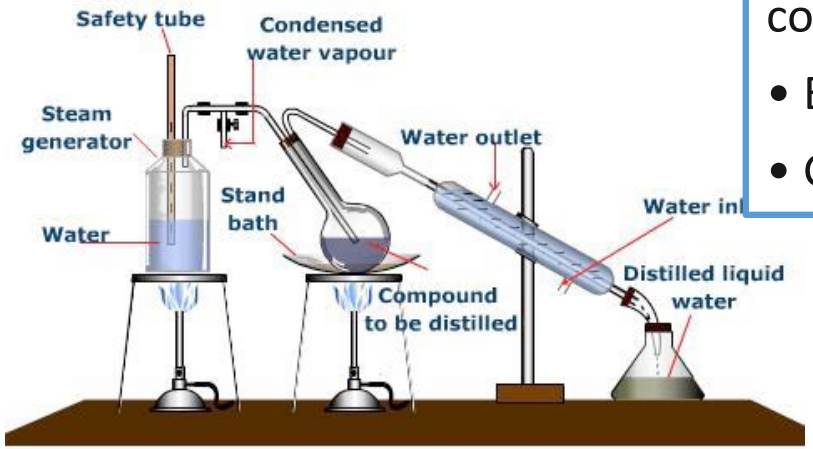
Essential Oils	Pectins	Polyphenols

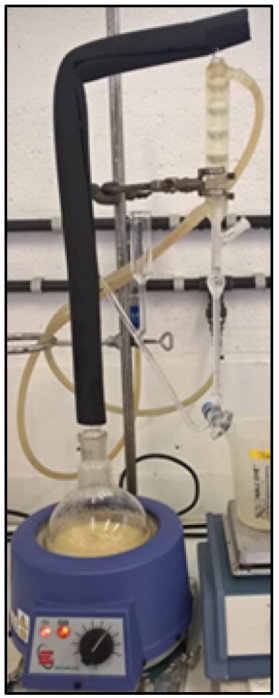
Extraction of Essential Oils



Distillation

- oldest and easiest methods
- Energy and time-consuming
- Partial degradation of compounds of interest
- Easy of implementation
- Good results!!





Distillation Vs $scCO_2$

LITERATURE:

$scCO_2$ performs slightly better:

- Energy & time
- Less oxidised compounds

Greener Improvements in Distillation of Essential Oils

- Design, steam efficiency
- Heat transfer

- Microwave
- Ultrasounds

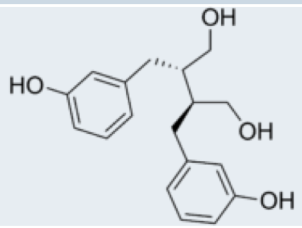
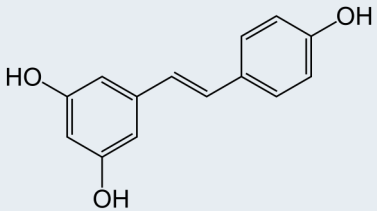
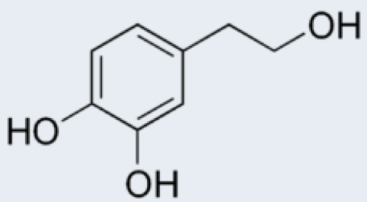
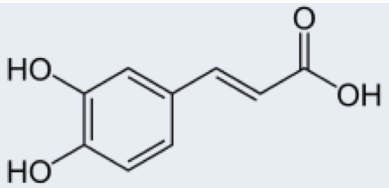
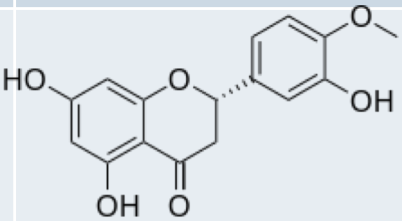
Greener Improvements in Distillation of Essential Oils

Ultrasounds	Microwave

- Less energy and time Vs standard distillation
- Less oxidation compounds
- Easy of implementation
- Good results!!

Polyphenols

- Secondary metabolites
- Different roles in vegetal sources: growth promoters, defence against predators…
- Nutraceutical properties
- Found in a wide range of products: cereals, berries, brasicas, wine, tea…

Lignans	Stilbenes	Phenolic alcohols	Phenolic acids	Flavonoids
Enterodiol	**Resveratrol**	**Hydroxytyrosol**	**Caffeic acid**	**Hesperetin**
				
Antiviral Antihypertensive	Anti-inflamatory Cancer	Antioxidant Blood lipid levels	Antioxidant Anti-inflamatory	Anti-inflammatory Cancer

Polyphenols

- Found as glycoside or as free form
- Properties and bioavailability depend on the food matrix
- Different chemistries, different solubilities…
- Sensitive to environmental factors (light, heat…)

AFTERLIFE Citromill Essential Oil Line Wastewaters:

Low yield of polyphenols extraction after Essential Oils recovery

Lysis of the polymer matrix is necessary

Enzymatic	Ultrasounds	Microwaves

Enzyme-assisted extraction

- Non-conventional & environmental friendly technology
- Becoming very popular
- Enzymatic treatment as a pretreatment of the raw material
- Specific & selective process

Mechanism: Degradation of cell walls and membranes

- Cell wall composition: polysaccharides (pectin, cellulose, hemicellulose…)
- Mode of action of the selected enzyme(s)
- Operational conditions:

Enzyme concentration	Enzyme to substrate ratio
Temperature & time	pH
Stirring	Particle size

Advantages

- Easy of test at laboratory scale
- Common food-grades enzymes work
- Low cost at small scale
- Mild conditions, enzymes can adapt to different environments

Limitations

- Large-scale application
- High costs for large volume
- Enzymatic behaviour at industrial scale
- Current availability of enzyme preparations

AFTERLIFE Citromill Essential Oil Line Wastewaters:

Promising results in initial tests with Pectinase 62L (P62) from Biocatalysts

ECOPEC project: ecological production of pectin from apple pomace and its use in organic jelly

Literature

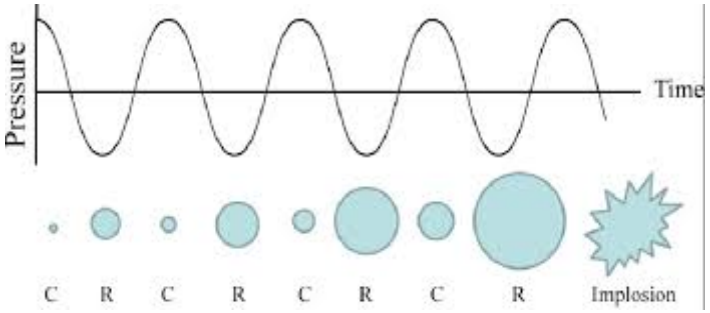
Target Compounds	Source	Yields	Enzymes	Ref
Pectin	Chicory root	34,6g/100g	Mixture (cellulases, pectinase and protease)	Panouillé 2006
Lycopene	Tomato peel	0,11g/100g	Cellulase Pectinase	Choudhari 2007
Carotene	Carrot pomace	6,4mg/100g	Pectinase and Celllase	Stoll 2003
Phenolics	Citrus peel	90-162mg GAE/100g	Mixture Cellulases	Li 2006
Phenolics	Apple skin	105mgGAE/L	Mixture Pextinex Smash, Celluclast and Sumizyme	Pinelo 2008
Phenolics	Grape pomace	6g GAE/L	Pectinase (Grindamyl)	Meyer 1998
Phenolics	Apple pomace	908mg GAE/100g	Comercial Pectinases	Zheng 2008

Ultrasounds

- Non-conventional & environmental friendly technology
- Easy to handle and implement
- Safe, economical and reproducible
- Possible coupling to other technologies

Mechanism: Acoustic Cavitation

- Collapse of gas bubbles as violent implosion
- Puntual/localized high Pressure & Temperature
- Shockwave induced damages in plant tissue
- Microjet impacts in the Surface of tissue
- Time and energy saving process



Literature

Target Compounds	Source	Yields	Conditions	Ref
Pectin	Grapefruit peel	27.4g/100g	HCl aqueous solution pH 1.5 U.S. probe, 66.7ºC, 27,95 min	Wang 2015
Phenolics, Tocopherol	Olive leaves	41 mg Oleuropein eq /100g oil	Olive oil as solvent U.S. bath, 16ºC, 45 min	Achat 2012
Phenolics	Wheat bran	312 mgGAE/100g	Ethanol 64% U.S. bath, 60ºC, 25 min	Wang 2008
Phenolics	Coconut shell	22442mg GAE/100g	Ethanol 50% U.S. bath, 30ºC, 15 min	Rodrigues 2008
Phenolics	Apple pomace	555mg Catequin/100g	Water U.S. bath, 40ºC, 40 min	Pingret 2012
Phenolics	Chicory	723 mgGAE/100g	Ethanol 37.5% U.S. probe, 60ºC, 9.2 min	Pradal 2016
Phenolics	Apple pomace	964mg Catequin/100g	Ethanol 50% U.S. bath, 40ºC, 40 min	Virot 2010

Ultrasound-Assisted Extraction Parameters

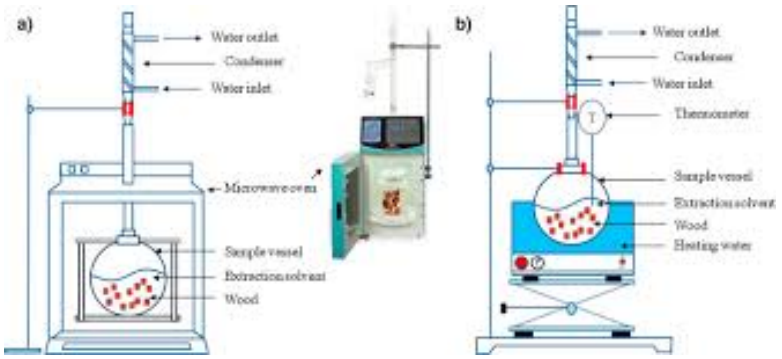
- Ultrasonic Bath Vs Probe
- Continuous sonication Vs Pulsed mode
- Operation Frecuency
- Amplitud
- Solvent
- Pretreatment
- Temperature
- Time

Microwave-assisted extraction

- Non-conventional & environmental friendly technology
- Easy to handle and implement
- Safe, economical and reproducible
- Heat sensitive compounds (where rapid heating and shorter time is necessary)

Effect of μwaves on molecules by ionic conduction and dipole rotation

- Non ionizing radiation to heat molecules
- Quickly heating of solvent
- Solvent-free process is possible

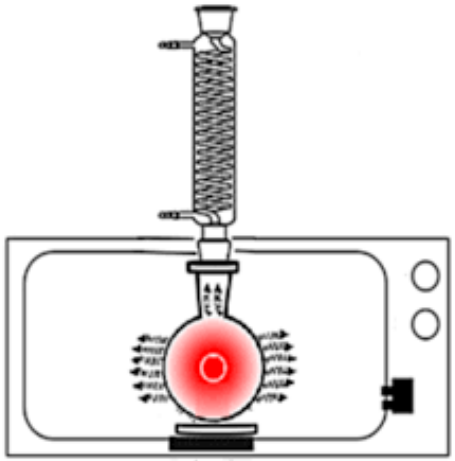


Literature

Target Compounds	Source	Yields	Conditions	Ref
Pectin	Papaya peel	25.4g/100g	HCl aqueous solution pH 1.8 512W; .140 sec	Prakash Maran 2014
Lycopene	Tomato leaves	13,6 g/100g oil	Hexane-ethyl acetate 400W	Ho 2015
Phenolics	Citrus peels	1220 mgGAE/100g	acetone 51% 500W, 122 sec	Nayak 2015
Phenolics	Mandarin peels	2320mg GAE/100g	Water 400W, 180 sec	Ahmad 2012
Phenolics	Peanut skin	14360mg GAE/100g	Ethanol 30% 90% of power, 30 sec	Ballard 2010
Phenolics	Myrtus leaves	16249mgGAE/100g	Ethanol 42% 500W, 62 sec	Dahmaune 2015
Phenolics	Potato waste	1100mg GAE/100g	Ethanol 60% 80ºC, 120 sec	Wu 2012

Microwave-Assisted Extraction Parametres

- Solvent type and volume
- Solid to solvent ratio
- Operation Power
- Temperature
- Time



PERSPECTIVES

Combination of Green Technologies	Bio-refinery Concept	Process Analysis

APPLICATION

"A project focused on the recovery technologies without establishing definite applications of the final product, is doomed to fail"

(Galanakis 2017)

THANK YOU !!

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