Interreg North-West Europe
AgriWasteValue
European Regional Development Fund

Recovery and enzymatic modification of bioactives metabolites from agricultural residues using green technologies: methodology and processes

AgriWasteValue mid-term event

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26th of January 2021

Preliminary screening of bioactives

Which bioactives in which concentration in which biomasses ?

Collection of samples

Grapes – *5 varieties*

Apple – *10 varieties*

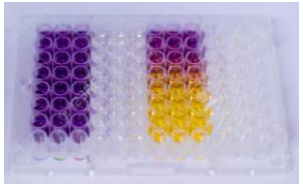
Pear – *3 varieties*

Bioactives extraction

Solvent of different polarities

Biological activities

Anti-oxidant capacities



Preliminary screening of bioactives

-High levels of polyphenols in EtOAc & EtOH70% extracts
-Important anti-oxidant activity of EtOH70% extracts

-**Phloridzin** as majority bioactive (10-15%)
(few differences between varities)

-High levels of polyphenols in EtOAc & EtOH70% extracts
-Important anti-oxidant activity of EtOH70% extracts

- **Resveratrol**, **Viniferin** (2-10%), **Catechin**, **Epicatechin**, (~ 1%)
(more differences between varities)

-High levels of in H_2O & EtOH70% extracts
-Important anti-oxidant activity of EtOH70%

Catechin, **Epicatechin**, **Chorogenic acid** (~ 1%)

Extraction methodology

Apple Trees
(10 varieties)

- *Jonagold*
- *Jonagored*
- *Braeburn*
- *Golden*
- *Gala*
- *Elster*
- *Novajo*
- *Bisquet*
- *Saint Remy C.*
- *Rouge Duret*

Classical maceration

Environmentally friendly methods

Accelerated solvent extraction (pressurized extraction)

Ultrasound assisted extraction

Comparison of extraction methods

PARAMETERS / METHOD	TEMPERATURE	DURATION	NUMBER OF CYCLES	RATIO (MASS/VOLUME OF SOLVENT)
ASE	120°C	10’	2	/
UAE	RT	30’	1	1/5
				1/10
		1h		1/5
				1/10
Maceration	RT	4h	1	1/5
				1/10
	50°C			1/5
				1/10

- Best extraction yields with **ASE**
- But **no better extraction method** to obtain phenols or antioxidant compounds

Study of the scaling-up

Lab-scale (20 gr)

Medium-scale (200 gr)

Semi-pilot scale (4500 gr)

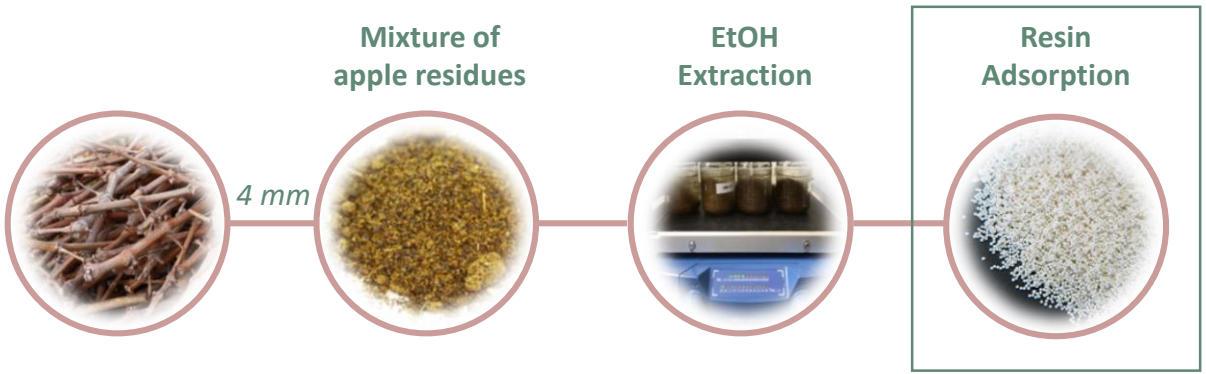
- Maceration using the following conditions:
  - Solvent: EtOH

Study of the scaling-up

	Lab-scale (20 gr)	Medium-scale (200 gr) – 1 cycle	Medium-scale (200 gr) – 2 cycle	Semi-pilot scale (4500 gr)
Extraction yields	5,4 %	11,3 %	2,6 %	13,2 %
Phloridzin content	15,6 %	19,7 %	13 %	20 %

- ❑ The extraction yield increased as we go to higher scale
- ❑ Moreover, phloridzin content increased also in higher extraction scale

Bioactive concentration



	Initial extract	Enriched extract
Extract mass recovered	100 g	47.3 g
Phloridzin content	20% →	49%
Phloridzin recovered in concentrated extract		100%

- Total recovery of the bioactive (Phloridzin)
- High concentrated extract in Phloridzin

Extracts characterization

	Initial extract	Enriched extract
Total Phenolic content	**160** mg EGA/g	**385** mg EGA/g
Anti-oxidant activity (DPPH)	**41** mg EGA/g	**75** mg EGA/g
Anti-oxidant activity (FRAP)	**0.46** mmol eq Fe(II)/g	**1.25** mmol eq Fe(II)/g

After resin adsorption

Extraction methodology

- Classical maceration
- Environmentally friendly methods
  - Accelerated solvent extraction (pressurized extraction)
  - Ultrasound assisted extraction

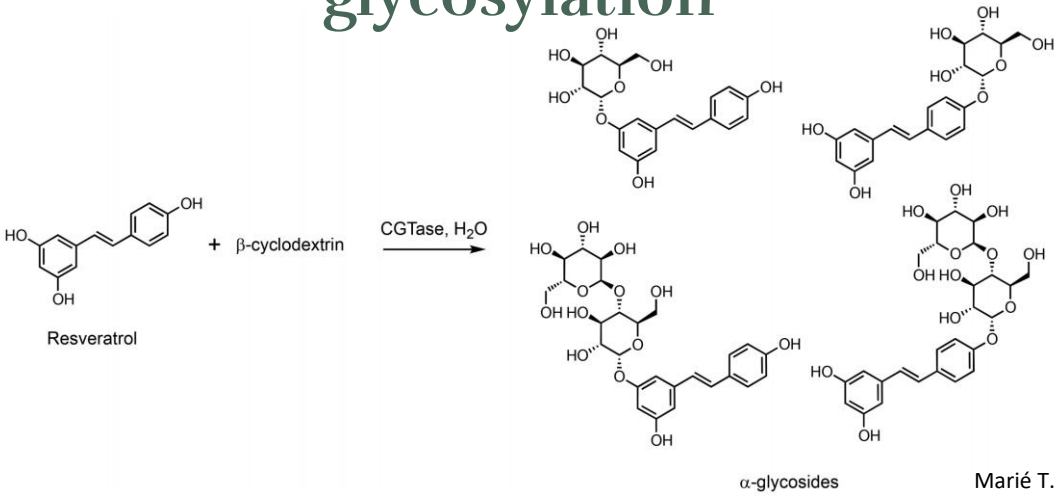
Objective : concentrated extract in **resveratrol** and **viniferine** (on-going)

Bioactive modification

Enzymatical or chemical

- Increase of bioactivities
- Modification of properties
- Increase of bioavailability

Bioactive modification : Enzymatic glycosylation



➢ Add glucose(s) on free hydroxyle groups of compounds

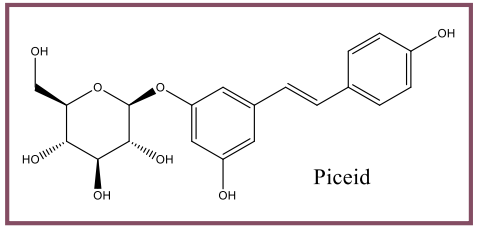
Marié T. *et al*. « Enzymatic Synthesis of Resveratrol α-Glycosides from β-Cyclodextrin-Resveratrol Complex in Water ». *ACS Sustainable Chemistry & Engineering* 6, nº 4 (2 avril 2018): 5370-80. DOI : 10.1021/acssuschemeng.8b00176.

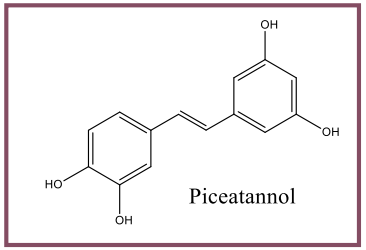
- ❑ New properties for resveratrol
- ❑ Increase the water-solubility and the bioavailability of compounds

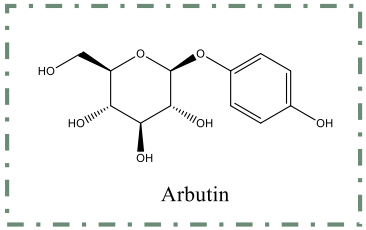
→ **use in cosmetic formulations**

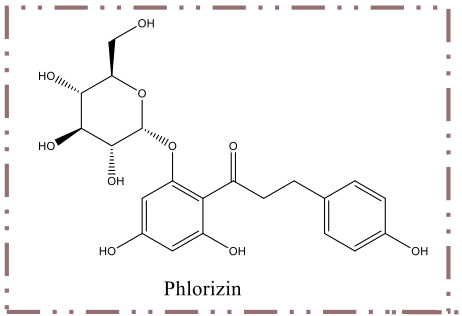
Bioactive modification : Application on other biomasses

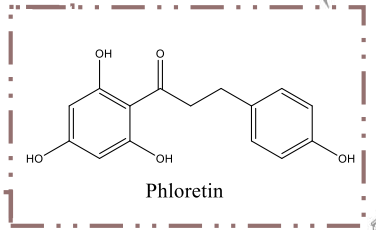
- Various molecules of vine, pear or apple tree wood can thus be glycosylated

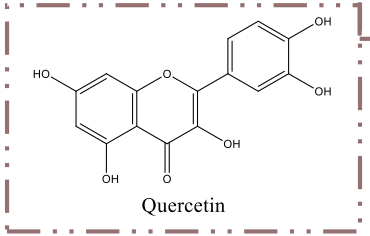












Recovery and enzymatic modification of bioactives

By-products

Mechanical Grinding

Extraction and enzymatic modification

Freeze-dried extracts

Cosmetic & nutraceutic actives

Extract residue valorization