

PRETREATMENT EFFECT ON ANTHOCYANIN EXTRACTION KINETICS FROM DIFFERENT WINE LEES

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Wine industry generates huge amounts of wastes and by-products, which are sources of high value compounds; including vine prunings, grape stalks, grape pomace and wine lees. For instance, anthocyanin (AC) extraction from grape pomace is a well-known process and several works can be found in literature. Similarly, wine lees (water-waste residues generated during maceration and fermentation steps) have been used to obtain tartaric acid or as a food supplement for animals. Additionally, the recovery of natural colorants (AC) from these waste streams has attracted much attention in the last years due to recent studies showed that the concentration of these colorants are 10 times higher than in grape skins. Thereby, the exploitation of these dregs could constitute to the development of new wine-related products and could also lead to a sustainable growth of the wine industry.

As a result, this work is focused on the development of effective green strategies and processes for the selective recovery of bioactive compounds from different wine lees of the first and second fermentation. Microwave (MW), sonication (S) and enzymatic (EM) pre-treatments followed by a solid-liquid extraction have been studied to intensify the extraction of AC in comparison with the conventional extractions. For conventional extraction kinetics, the parameters studied were the solvents ratio, the solid:liquid ratio (from 0.1-0.025 g/mL) and extraction temperature (25, 35 and 45°C). Furthermore, process parameters for each type of pretreatment were also investigated such as residence time for MW and S or substrate:enzyme ratio in EM approach. Final results shows that the best behavior obtained for the solid-liquid extraction takes place when a 50:50 (v/v) EtOH:H₂O mixture, a RS-L of 1/10 (g/mL) and a temperature of 25°C are used. Results showed that sonication pretreatments only reduced the extraction time, achieving a final AC concentration similar to the conventional process. On the other hand, enzymes consumed almost the same time but the AC concentration increased approximately in 40% (w/w). Conversely, MW pretreatments seemed to be the most promising pretreatment due to a yield improvement (151% w/w) and a substantial shorter extraction time (90s against 10 minutes).



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WINE LEES REVALORIZATION

Pretreatment effect on anthocyanin extraction kinetics from
different wine lees

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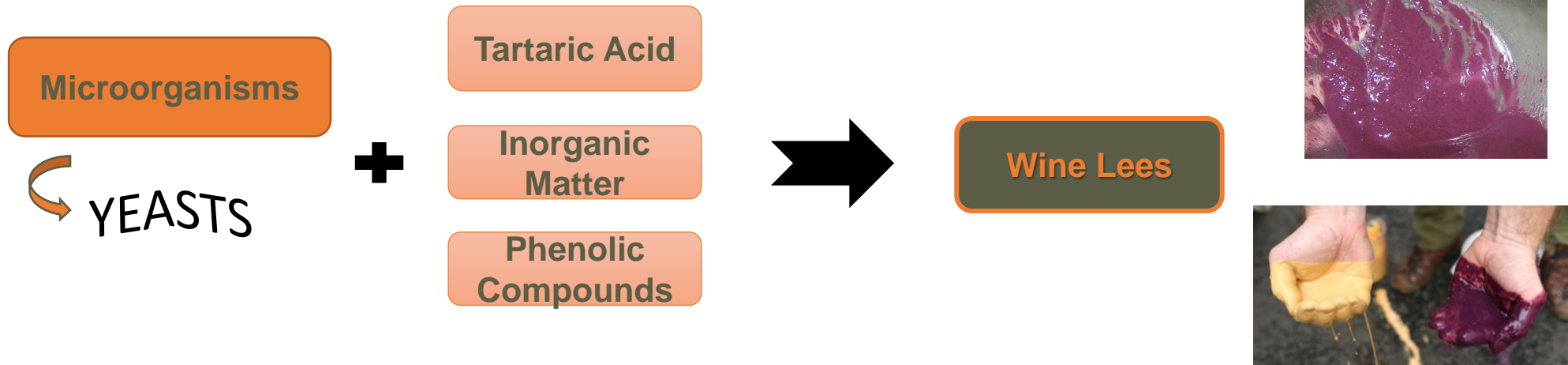
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Thursday, 8 June 2017

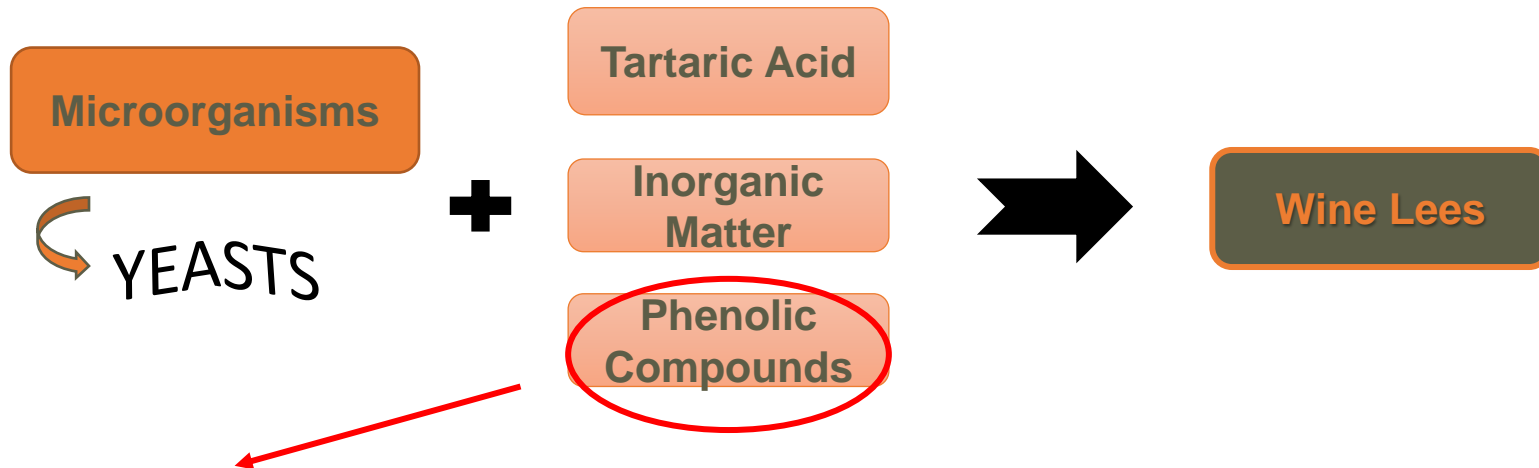
Introduction

- ✦ Winery production is one of the most important agricultural activities throughout the world: → 60 million metric tons of grapes are generated per year over the world.
- ✦ Wine making produces different residues → Wine lees are semi-solid residues generated during fermentation and maceration steps of red and white wine production.



Introduction

- ✦ Winery production is one of the most important agricultural activities throughout the world: → 60 million metric tons of grapes are generated per year over the world.
- ✦ Wine making produces different residues → Wine lees are semi-solid residues generated during fermentation and maceration steps of red and white wine production.



- ✦ Anthocyanins → main phenolic in red wine and grapes

Antioxidant, antimicrobial, anti-inflammatory and anticarcinogenic activities



Objectives

- ✦ Recovery of high added compounds from wine lees:
 - Maximization of anthocyanin extraction.

- ✦ Different extraction procedures:
 - Study of the extraction kinetics of **anthocyanins** and the influence of main parameters.
 - Applications of some pre-treatments before a S-L extraction:
 - ✦ Ultrasounds
 - ✦ Microwaves
 - ✦ Enzymes



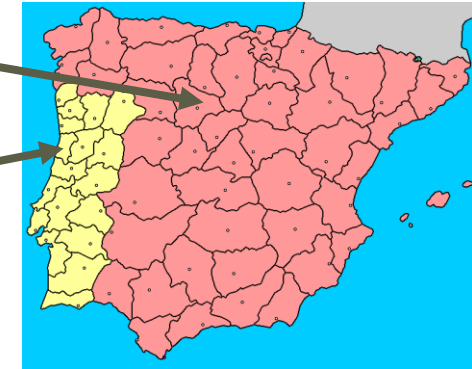
Materials and methods

✦ Three types of wine lees:

- **Two Ribera del Duero wine lees, *Matarromera winery*.**

✦ Wine lees from first and second fermentation

- **Porto wine lees, *Sogrape winery***



✦ To preserve the lees (mixture of solids and wine):

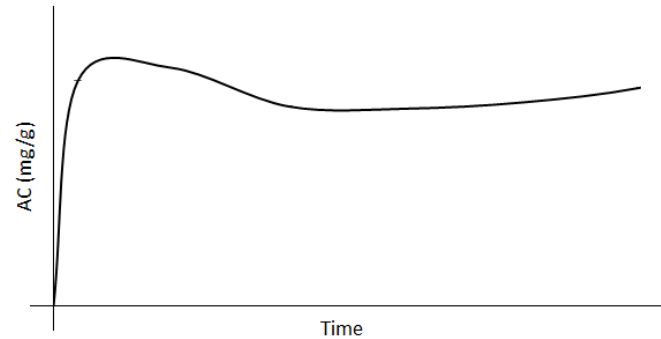
→ Centrifuged

→ Freeze-dried



Materials and methods

★ Extraction procedures:



pH differential method

AC Maximization

• Conventional **anthocyanin** extraction kinetics curve.

Fixed Conditions

- 300 rpm,
- pH = 2.6
- t = 120 min

Parameters

- Solid-liquid ratio (g/mL): 0.100, 0.050, 0.035, 0.025
- Solvent (%EtOH): hydro-alcoholic mixtures (0, 25, 50 and 75)
- Temperature: 25, 35 and 45°C.
- Time (min)

S-L
extraction

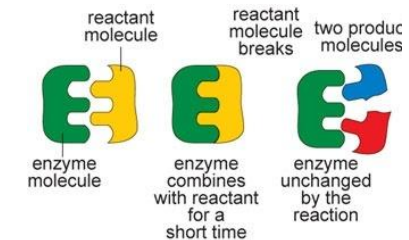


Materials and methods

★ Extraction procedures:

Pre-
treatment

- Microwaves
- Ultrasounds
- Enzymes



S-L
extraction

- Best conditions from the kinetic study.



Fixed Conditions

- 300 rpm,
- pH = 2.6
- t = 120 min

Parameters

- Solid-liquid ratio (g/mL): 0.100, 0.050, 0.035, 0.025
- Solvent (%EtOH): hydro-alcoholic mixtures (0, 25, 50 and 75)
- Temperature: 25, 35 and 45°C.
- Time (min)



Materials and methods

★ Microwave pre-treatment:

Three main variables:

- solid-liquid ratio (g/mL): 0.10, 0.15, 0.20
- solvent mixture (% H₂O): 10, 55, 100
- time of microwaves (s): 30, 60, 90



Total of 17 experiments

Statistical surface response

- three levels (-1, 0, 1)
- triplicate of the central point



★ Ultrasounds pre-treatment:

Two main variables:

- amplitude (%): 10, 50, 100
- time of ultrasounds (s): 60, 90, 120



Total of 11 experiments

Statistical surface response

- three levels (-1, 0, 1)
- triplicate of the central point



★ Enzymatic pre-treatment:

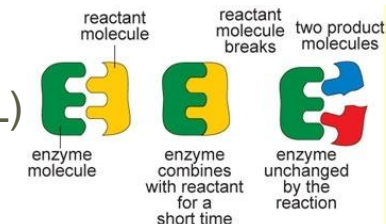
Variable:

- Incubation time (min): 5, 15, 30, 60
- two types of enzymes: Glucanex[®], Mannaway[®] (Novozymes)



Fixed parameters:

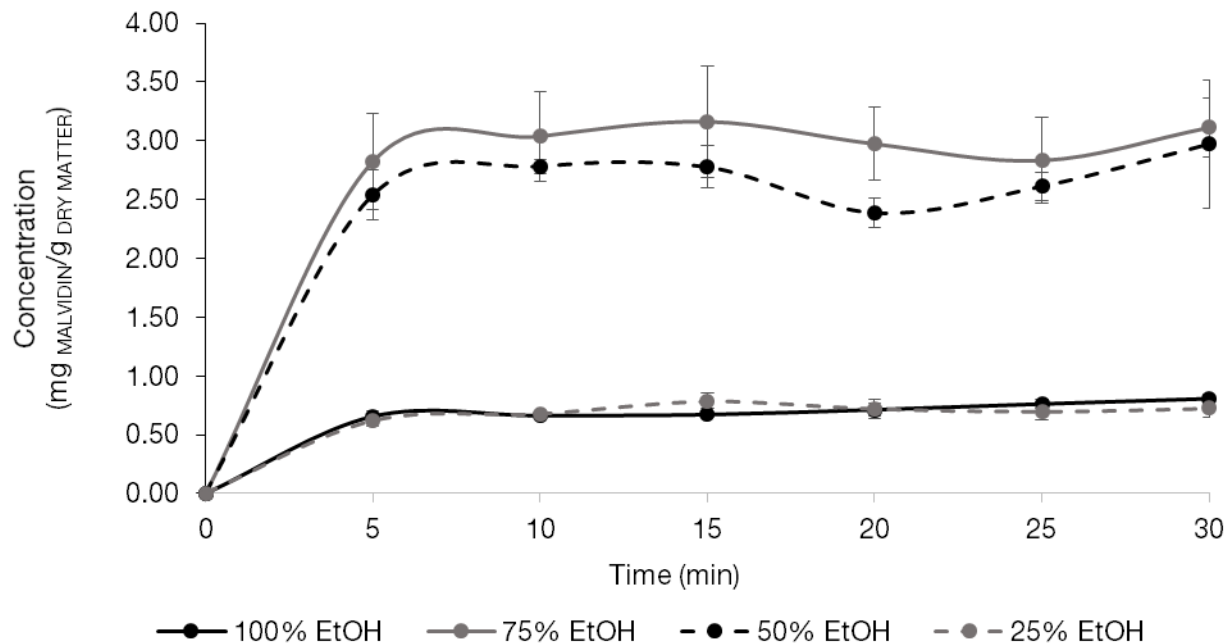
- 300 rpm
- solid-liquid ratio (1/10 g/mL)
- temperature (35°C)
- pH=5



Results

★ Solid-liquid extraction: Kinetic extraction curves

Parameters selected as the best for anthocyanin extraction



- R_{S-L} (g/mL): **0.10**
- Solvent: **50:50 EtOH:H₂O (% vol.)**
- T^a : **25°C**
- Time (min): **10-15**

Anthocyanin Conc.: (mg_{MALVIDIN}/g_{DRY MATTER})

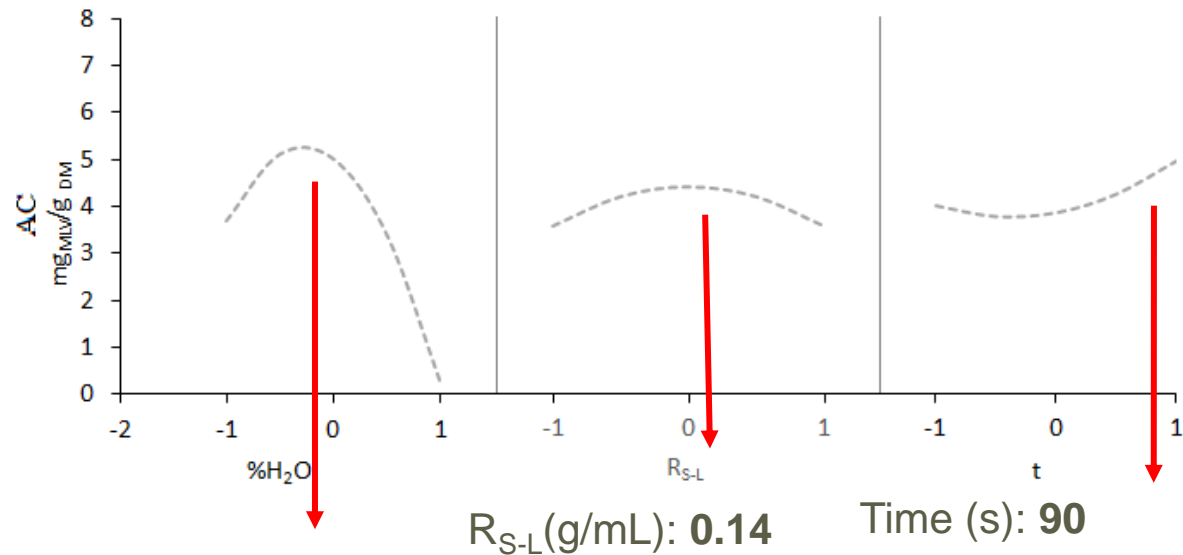
- ◆ 1st Ferm.: 3.04 ± 0.03
- ◆ 2nd Ferm.: 2.09 ± 0.38
- ◆ Porto lees: 2.29 ± 0.15



Results

★ Microwave pre-treatment

Main effect diagram of each variable for AC



Solvent: **40:60 EtOH:H₂O (% vol.)**

$$AC = 5.0166 + 0.624926 \cdot t - 0.00311123 \cdot R_{SL} - 1.74735 \cdot \%H_2O + 0.818187 \cdot t^2 - 0.0485444 \cdot t \cdot R_{SL} - 0.448549 \cdot t \cdot \%H_2O - 0.980728 \cdot R_{SL}^2 + 0.060261 \cdot R_{SL} \cdot \%H_2O - 3.07077 \cdot \%H_2O^2$$

Percentage absolute deviation: 12%

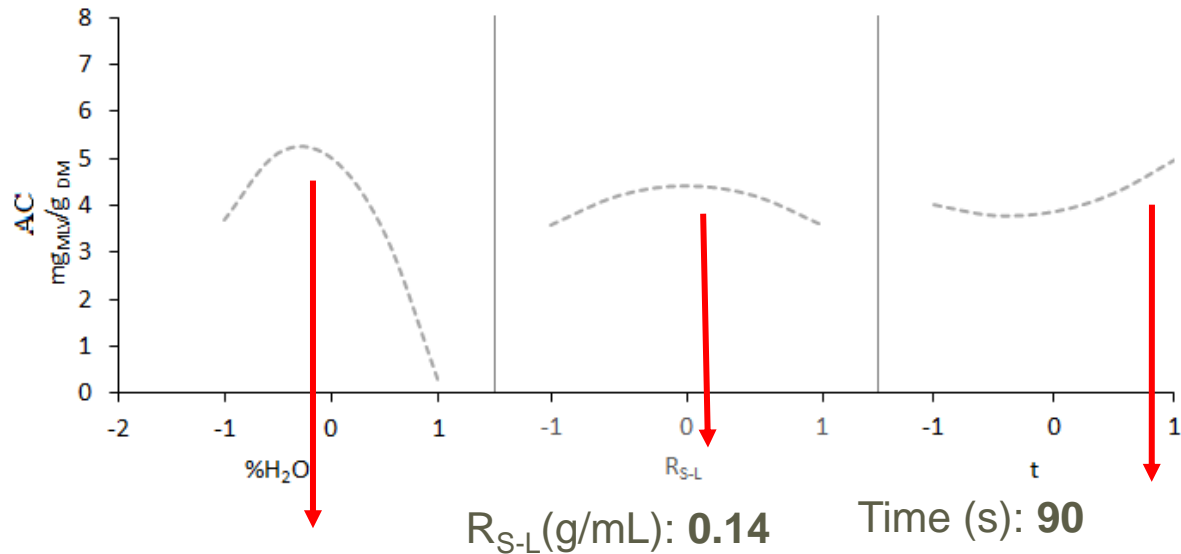
R² = 97,73%



Results

★ Microwave pre-treatment

Main effect diagram of each variable for AC

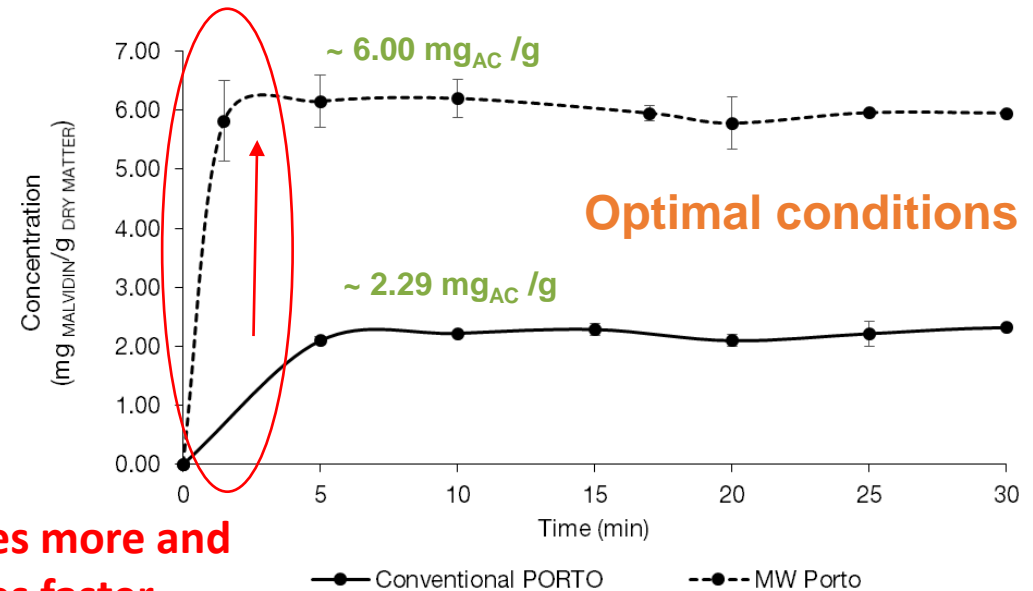


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Percentage absolute deviation: 12%

$$R^2 = 97,73\%$$



**3 times more and
5 times faster**

Figure: comparison between the S-L extraction and MW pre-treatment.



Results

★ Ultrasounds pre-treatment

After performing the sequence of experiments → no variations on final AC

- **AC AVERAGE** ($\text{mg MALVIDIN/g DRY MATTER}$): 3.30

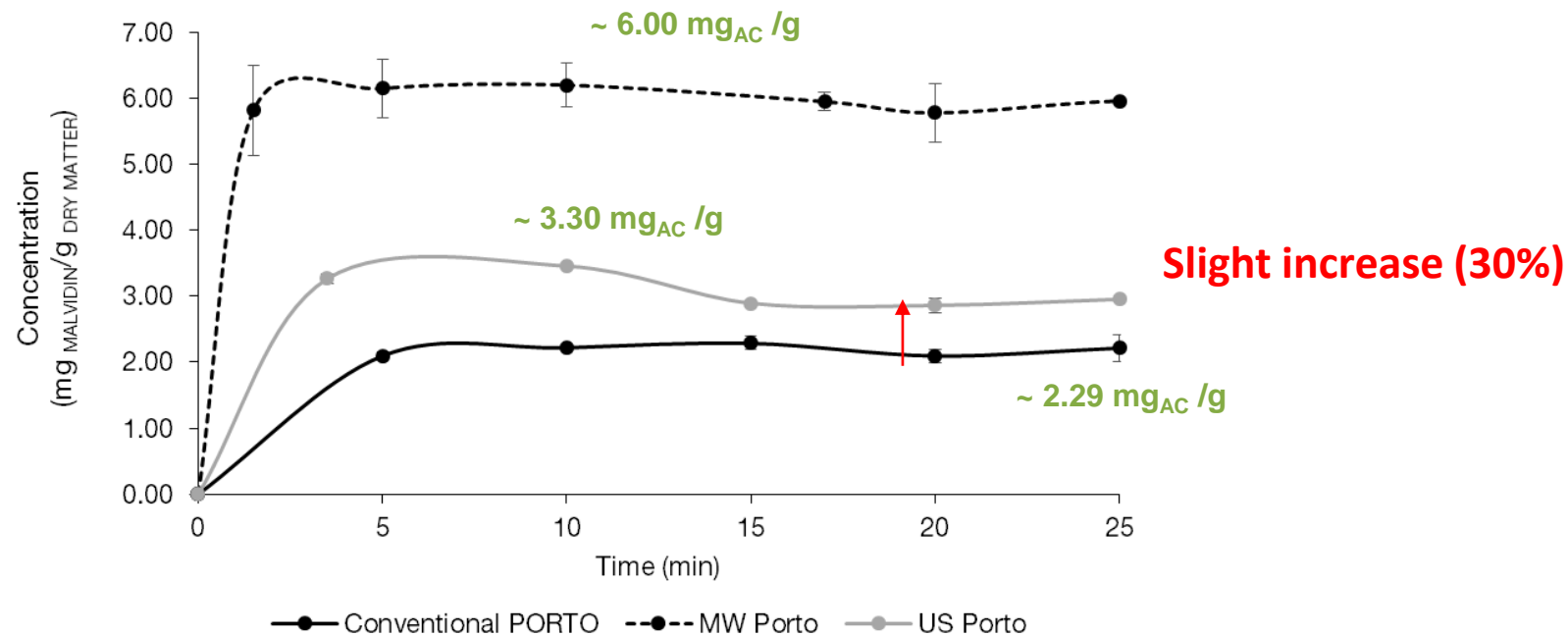


Figure: comparison between the S-L extraction, MW and ultrasounds pre-treatments.



Results

★ Enzymatic pre-treatment

- Incubation time (5, 15, 30 or 60 min) in H₂O → Increment of 10% using 60 min instead 5 min

→ No significant differences between both enzymes

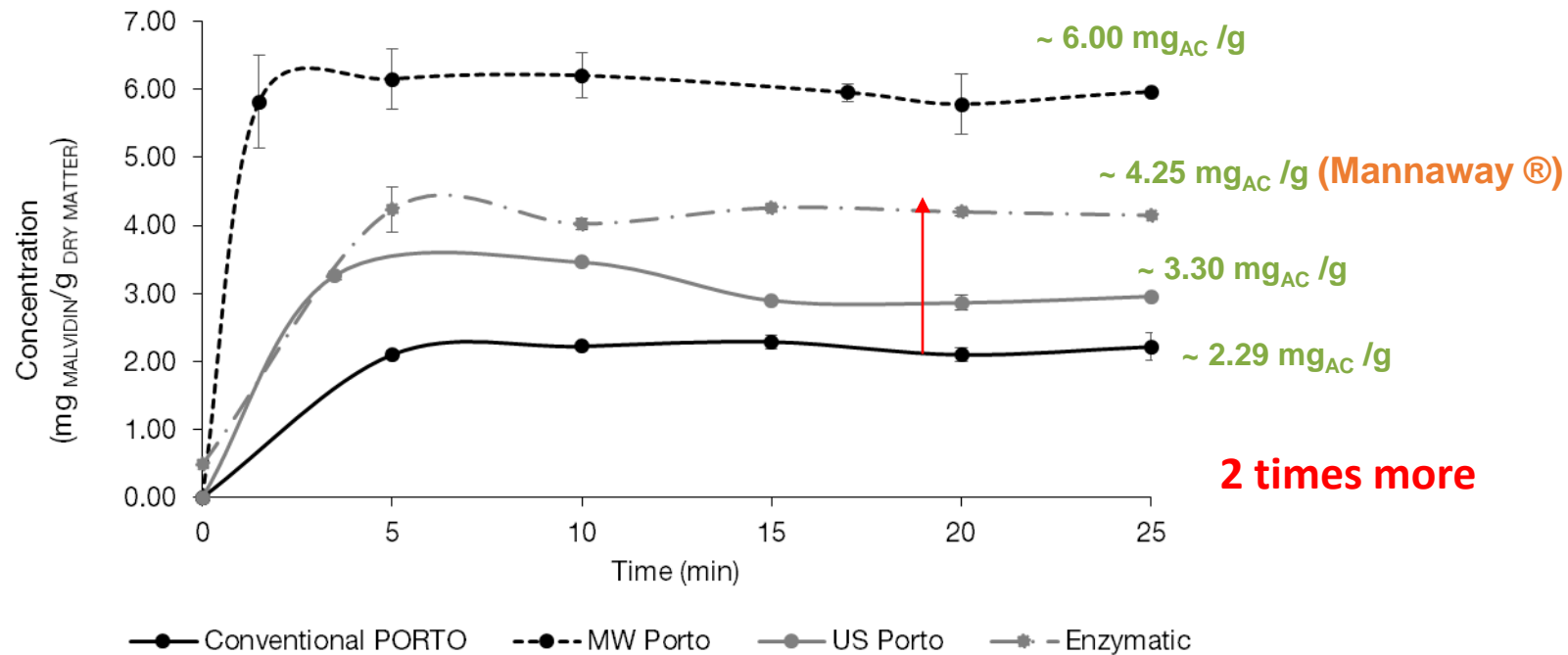


Figure: comparison between the S-L extraction, MW, ultrasounds and enzymatic pre-treatments.



Conclusions

★ Wine lees are a suitable source of polyphenols → 2.29 ± 0.15

★ Solid-liquid extraction best conditions:

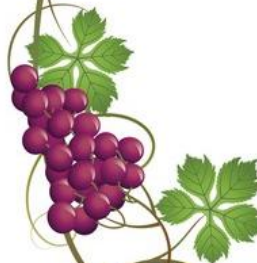
- R_{S-L} (g/mL): **0.10**
- Solvent: **50:50 EtOH:H₂O (% vol.)**
- T^a : **25°C**
- Time (min): **10-15**

★ Use of pre-treatments → enhance anthocyanin extraction

→ MW increases AC **three times (6mg/g)**. Reduces time from 15 min to **90 s**.

→ US **slightly** increases AC (30%). Reduces time from 15 min to **5 min**.

→ Enzymes increases AC **twice (4.5mg/g)**. **5 min** needed for incubation time + **10 min** extraction



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Thanks for your attention



WineSense



Ask A Question

