
# Effective production of bioactive phenolic compounds from olive stones

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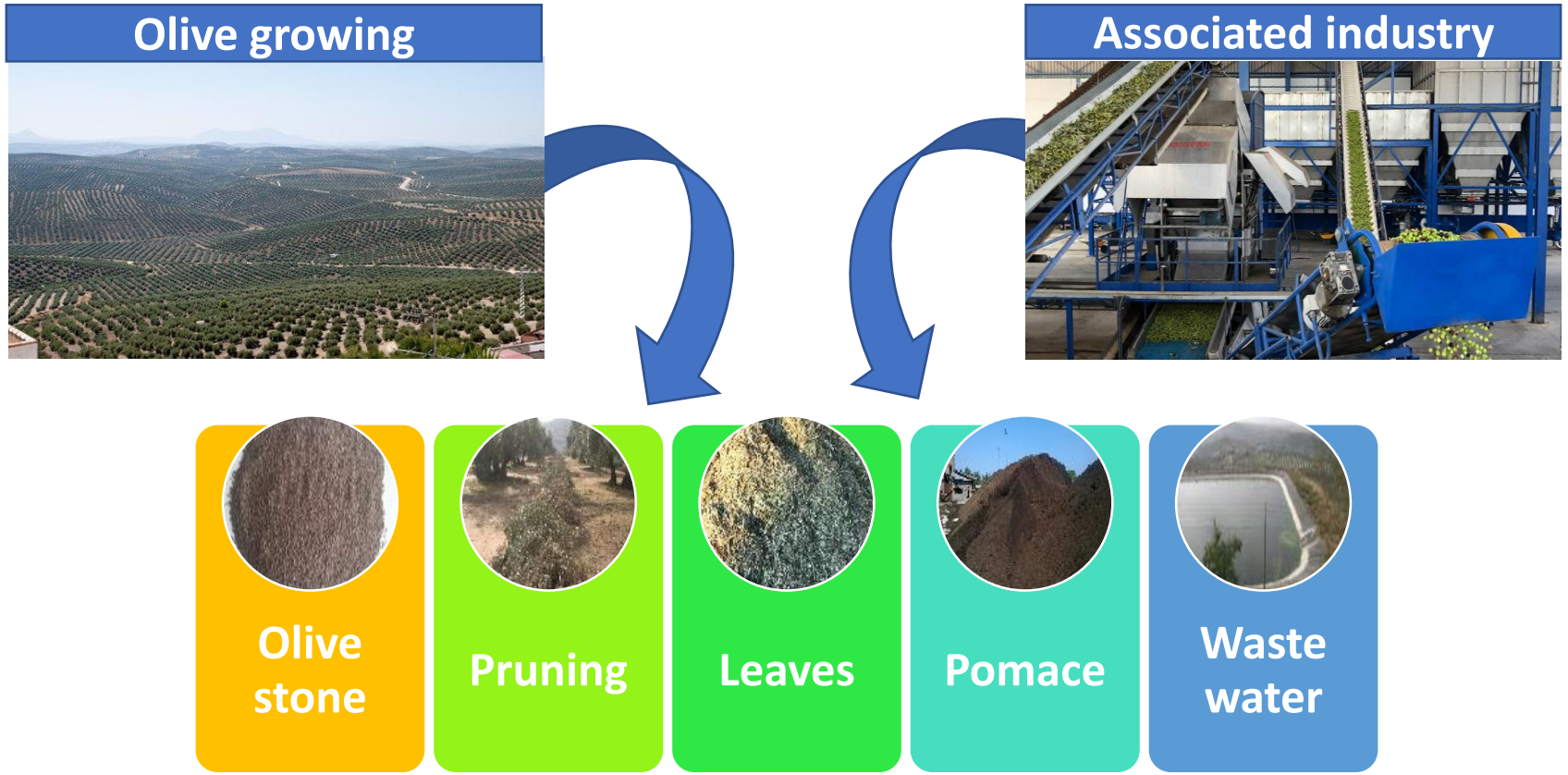
# Introduction

Olive growing and the associated industry generate a large amount of waste.



# Introduction

6 million tonnes/year of olives stones in Spain

The olive stone accounts for 10% of the weight of the olive

600,000 tonnes of olive stone per year

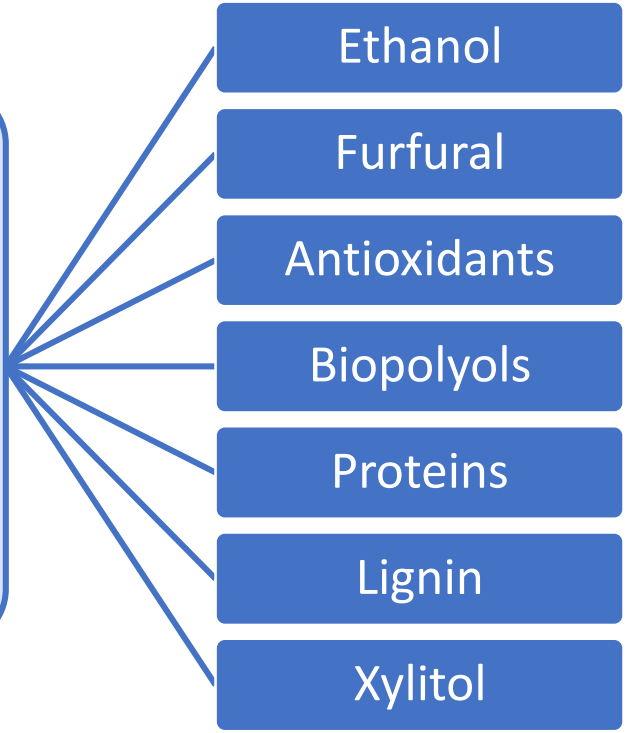
Olive stones (OS) are a by-product generated in the olive oil production process, obtaining an average of 600,000 tons of OS/year.

# Introduction

Olive stones:

- Renewable and abundant lignocellulosic biomass.
- High concentration of sugars.
- Centralised location in mills and associated industries.

It is an ideal material to obtain high added value products in the **BIOREFINERIES** concept:




**Antioxidants** present potential health benefits and applications for the pharmaceutical and food industries.

# Objective

The aim of this work is the valorization of the liquor obtained after a two-stage process, first acid stage followed by an organosolvent stage, for its use as a biosource of preservatives and non-synthetic additives for the food industry.

# Materials and methods

## BIOMASS CHARACTERIZATION

**OLIVE STONES**

**METHODOLOGY**

NREL
NATIONAL RENEWABLE ENERGY LABORATORY

- Sugars
- Extractives
- Acid Insoluble Lignin
- Acid Soluble Lignin
- Acetyl gropus
- Ash

# Materials and methods

**OLIVE STONES**

**ACID HYDROLYSIS**

**Box-Behnken design**
Time → 60 minutes
Temperature →128 °C
Biomass loading → 33%
$H_2SO_4$ concentration →10.55%
(g acid/100 g biomass)

**Solid**

**Liquid**

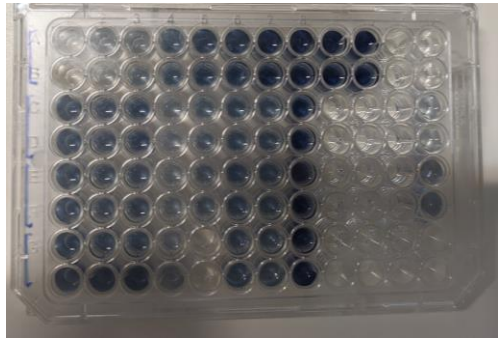
**ORGANOSOLV DELIGNIFICATION**

Ethanol:water → 50:50 (w/w)
Biomass loading → 1/6 solid/liquid (w/w)
Temperature → 140-190 °C
$H_2SO_4$ concentration → 0-100mM
Time → 0-150 min

**Liquid**

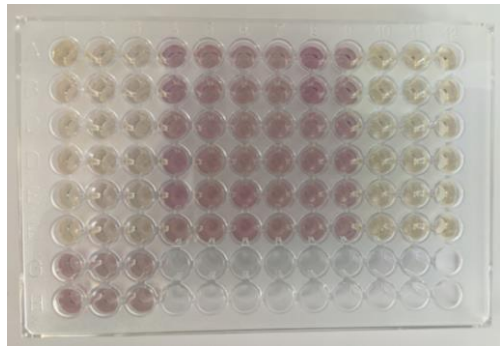
**Solid**

**TOTAL PHENOLIC CONTENT (TPC)**



**Folin-Ciocalteu**

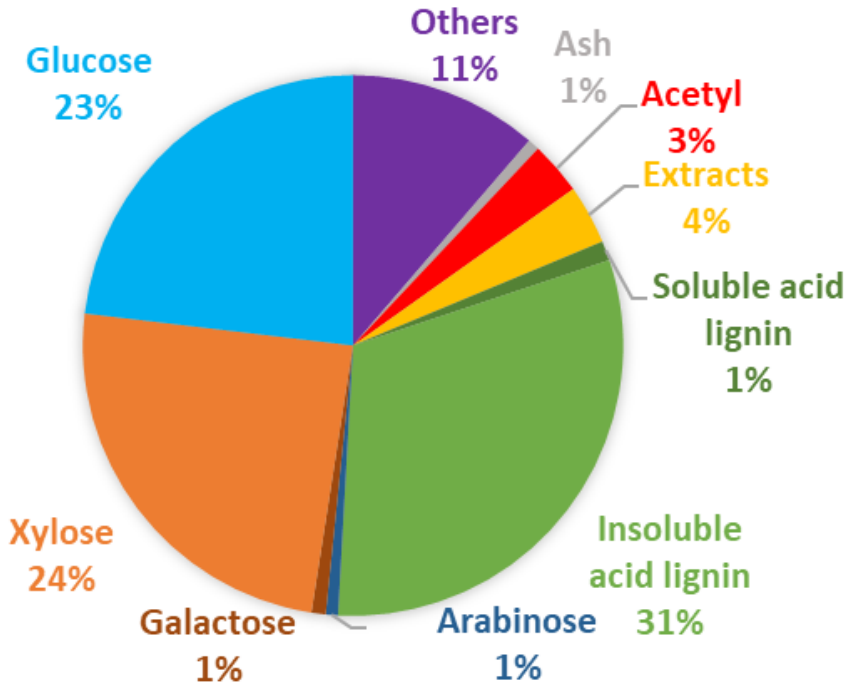
**ANTIOXIDANT ACTIVITY**



**DPPH**

# Results

## OLIVE STONES COMPOSITION




- Olive stones is a lignocellulosic material composed mainly of cellulose, hemicellulose and lignin.
- Xylose is the main hemicellulosic sugar.

# Results

## TWO-STAGE EXPERIMENTAL

### FIRST ACID STAGE

- Most of the hemicelluloses are solubilized in the first acidic stage.

### SECOND ORGANOSOLV STAGE

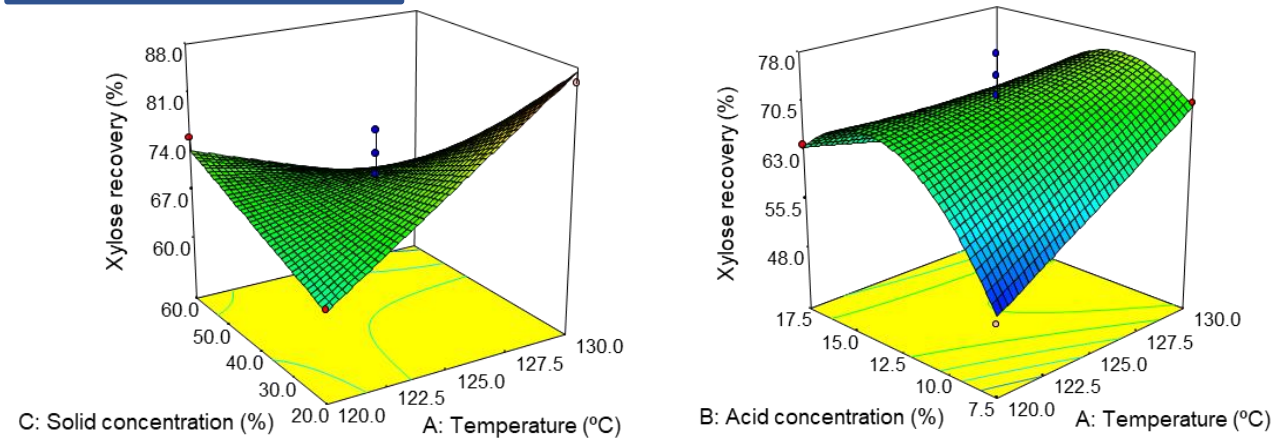
- In the second organosolv stage, most of the lignin is removed, resulting in a cellulose-rich solid.
- The liquor obtained in the second organosolv stage is enriched in phenolic compounds and antioxidants.

# Results

**In the first acidic stage, the aim is to maximise xylose recovery.**

## OPTIMIZATION OF THE EXPERIMENTAL CONDITIONS OF THE FIRST ACIDIC STEP

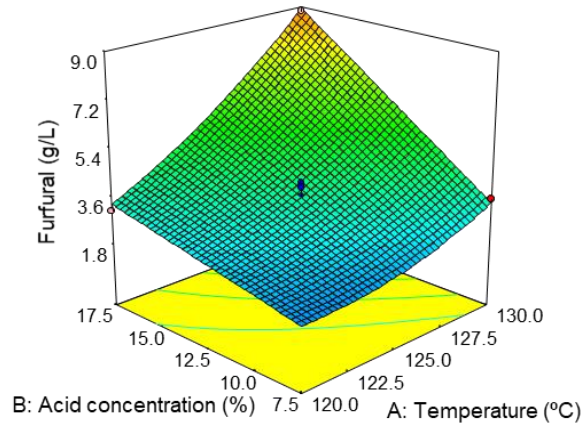
### Xylose recovery




- The pretreatment temperature had a positive influence at low solid loading on the xylose recovery but at high solid loading, its influence was negative.
- The temperature at low acid concentration had a positive influence on xylose recovery, while at the highest acid level this influence was negative.

### Furfural

The formation of furfural is indicative of xylose degradation.




When increased severity (high temperature and acid concentration), the concentration of furfural increases, and thus increasing xylose degradation.

**Optimal conditions**

- Temperature → 128ºC
- Acid concentration → 10.5%
- Solid concentration → 33%

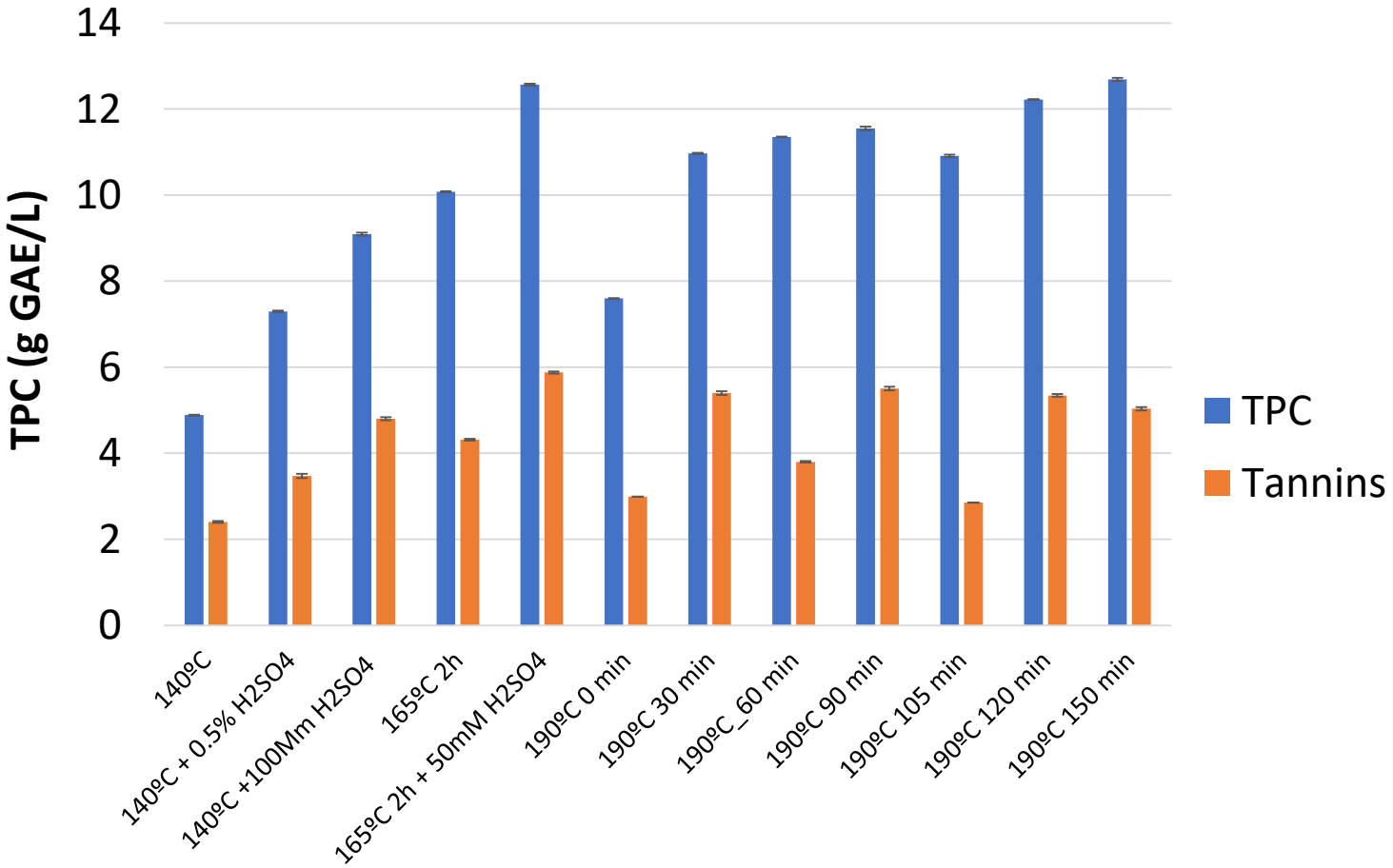
Pretreatment liquor:

- Xylose concentration: 60 g/L
- Xylose recovery: 76%

# Results

**In the second organosolv stage, the aim is to maximise phenolic compounds and antioxidants recovery.**

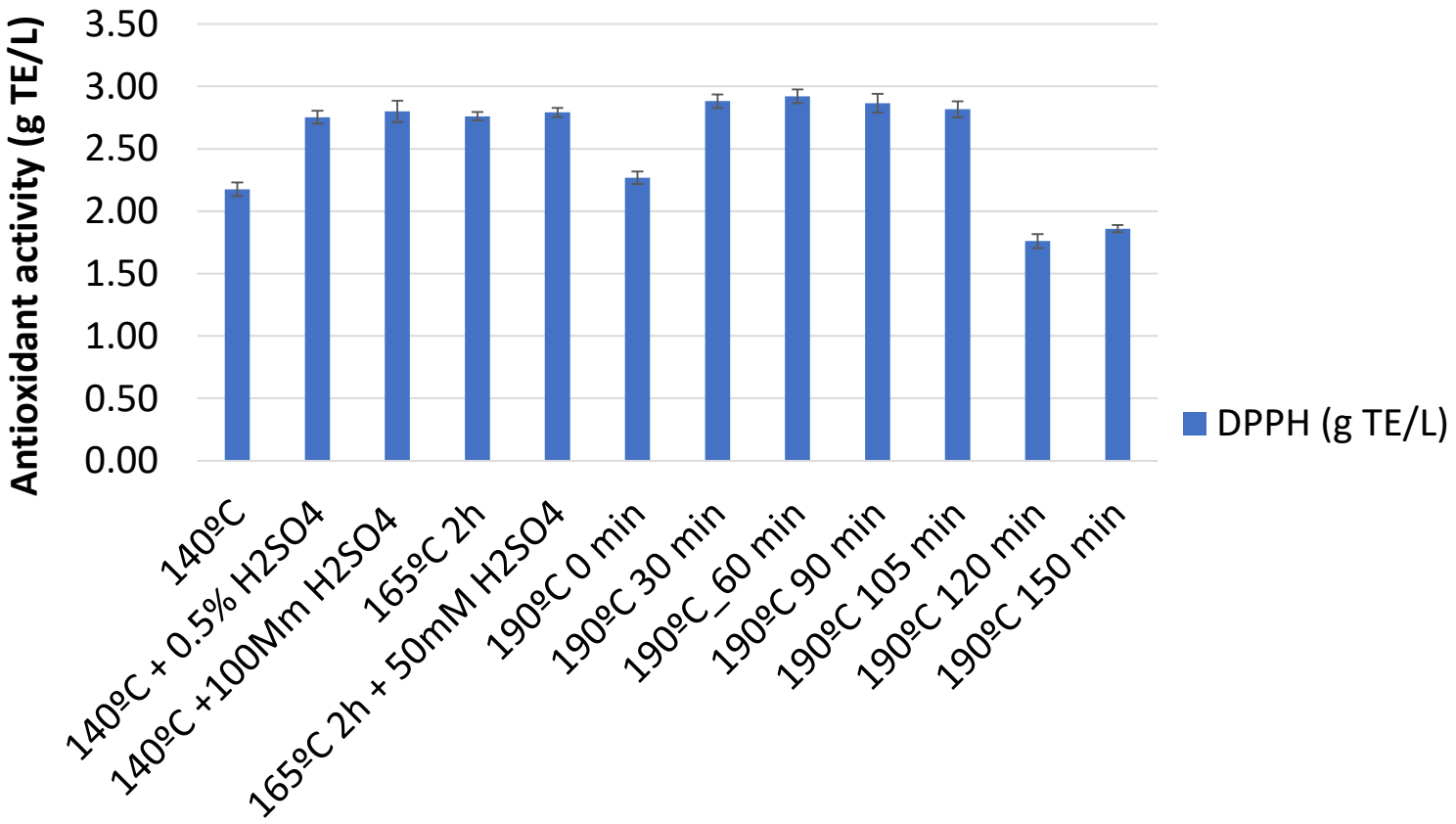
## TOTAL PHENOLIC CONTENT OF THE ORGANOSOLV LIQUID




- The phenols concentration measured in the liquors is between 5 and 12 g GAE/L.
- Tannins between 2 and 6 GAE g/L. Tannins are non-synthetic preservatives and additives.
- It implies a phenols yield of 7 GAE g/100 g of processed material, being in the range of those obtained from other vegetable sources.
- The highest concentrations of phenols and tannins are obtained in the experiments performed at 165ºC and 50 mM $H_2SO_4$ and at 190 °C in those with longer reaction time.

# Results

## ANTIOXIDANT ACTIVITY OF THE ORGANOSOLV LIQUID




- No major variations were observed in the concentrations obtained in the different experiments.
- The highest concentrations are found in the experiments at 190 °C and with times between 30 and 105 minutes, reaching almost 3 g TE/L.

# Conclusions

- The first acidic stage is optimal for xylose recovery.
- The liquor obtained after organosolv pretreatment of olive stones can be also valued as a bio-source of non-synthetic preservatives and additives for the food industry.

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