Recovery of polyphenols from agro-industries wastes: preliminary extraction studies

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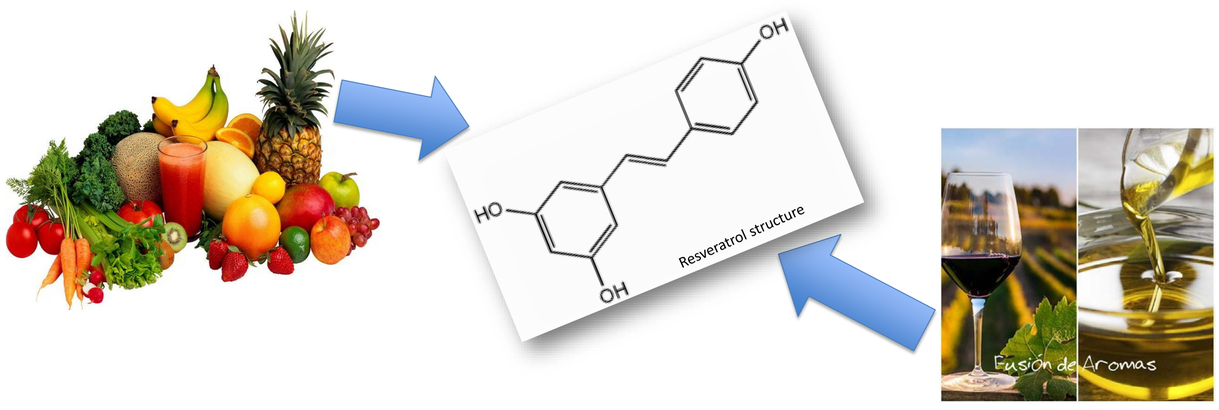
INTRODUCTION

Wastes generated during production of agro-food products, such as, fruits and vegetables juices, olive oil or wine are rich in bioactive compounds like polyphenols. These biocompounds have antioxidant properties and can be used in many valuable applications in the pharmaceutical, cosmetic and food industries, and thus their recovery from agro-food wastes is interesting (Kammerer *et al.*, 2014).

To speed up and/or to increase the efficiency of the polyphenols extraction, extraction techniques using supplementary energy can be advantageous (Rosello-Soto *et al.*, 2015; Souilem *et al.*, 2017). In this work the application of ultrasound assisted extraction (UAE) and microwave assisted extraction (MAE) have been studied for the extraction of polyphenols from 4 agro-food industry wastes, *i.e.* orange peel and seeds, spinach leaves, olive wastes and lees.



MATERIALS AND METHODS

To optimize UAE and MAE, factorial experimental designs were applied. For UAE the factors considered were % ethanol and % HCl for a fixed extraction time of 30 minutes. For MAE, % ethanol, time and also temperature were considered.

Table 1. Experimental design for UAE and MAE conditions.

Technique	Factor	Level
UAE	Ethanol (% v/v)	40, 60, 80
	HCl (% v/v)	0.0, 0.1, 0.5
	Time (min)	30
MAE	Ethanol (% v/v)	0, 40, 60
	Temperature (ºC)	60, 90, 120
	Time (min)	5, 15, 30

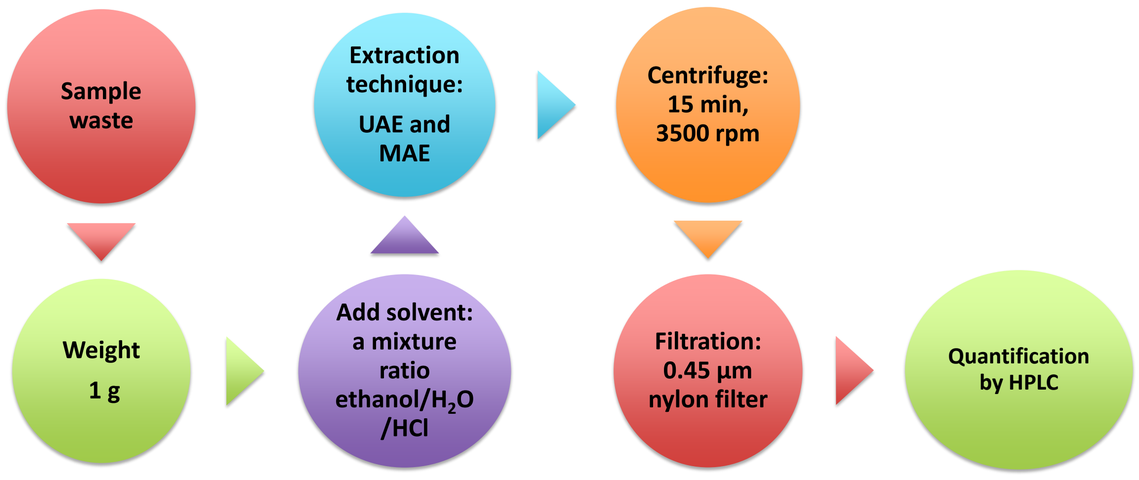


Figure 1 Polyphenols extraction process from orange peel and seeds, spinach leaves, olive wastes and lees.

The extracts were further analyzed by high performance liquid chromatography (HPLC) with diode array detection at the following conditions:

- Kinetx C18 column (100 mm x 4.6 mm, 2.6 µm)
- Mobile phases: A: 0.1% formic acid in water, and B: acetonitrile
- Gradient elution mode
- Flow rate: 0.4 mL/min
- Injection volume: 5 µL
- Run time: 39 min
- Detection at 280 nm (Raja *et al*., 2014)

HPLC Agilent 1100

RESULTS AND DISCUSSION

The maximum polyphenol extraction was obtained by a mixture containing ethanol/H_2O/HCl 60/39.9/0.1 (v/v/v). This mixture provided satisfactory results in the extraction of polyphenols from the assayed matrices, using either UAE (during an extraction time of 30 minutes) or MAE (establishing an extraction time of 15 minutes and a temperature of 90ºC).

Overall, the UAE process provides similar results to MAE, except for the orange matrix. In this case, total polyphenol extraction efficiency using MAE was about 4 times higher than that obtained by UAE.

On the other hand, some polyphenols degradation in lees and spinach extracts, when using MAE above 90°C, was observed. As a matter of example, Figure 2 shows the chromatograms of spinach extracts obtained by MAE at 90°C and 120°C.

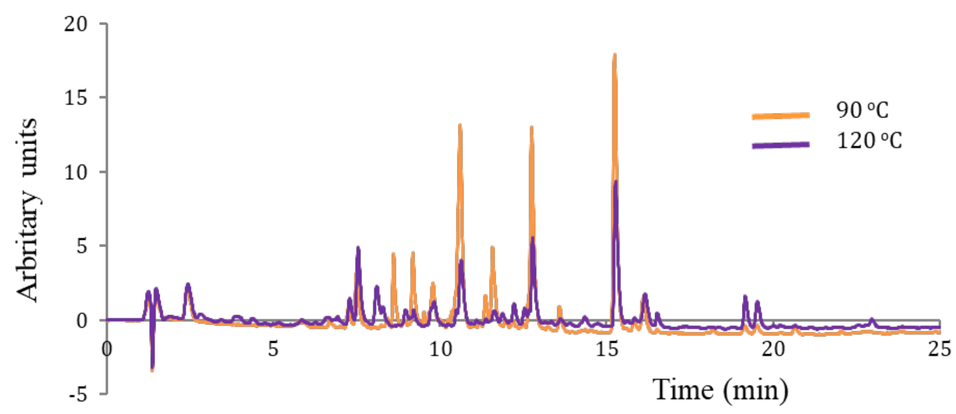


Figure 2. HPLC-UV chromatograms of spinach leaves extracts obtained by MAE at two temperature conditions. Extraction solvent: Ethanol/H_2O/HCl 60/39.9/0.1 (v/v/v). Extraction time: 15 min.

CONCLUSIONS

A mixture containing low acid concentration (0.1%) and 60% ethanol, has shown good performance for polyphenols extraction. For spinach waste, olive pomace, and lees UAE technique provides similar results than MAE, whereas for orange waste MAE shows better extraction results.

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