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Valorization of apple tree wood residues by polyphenols extraction: Comparison between conventional and microwave-assisted extraction

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ABSTRACT

For the first time, the characterization of antioxidant activity and phenolic profile of apple tree (*Malus domestica*) bark, core and roots was carried out. Phenolic compounds were extracted from the Belgium apple tree wood residues collected at two seasons, namely summer 2015 and winter 2016, using conventional (CE) and microwave-assisted extraction (MAE) techniques. For each extraction technique, the influence of the most important operational parameters, namely solvent composition, extraction time and temperature, on the total phenolic and flavonoid content, and antioxidant activity by the 2,2-diphenyl-1-picrylhydrazyl radical scavenging activity (DPPH-RSA) and ferric reducing activity power (FRAP) assays were optimized. The phenolic profile from the obtained extracts was also characterized by high-performance liquid chromatography with photodiode array detection (HPLC-PDA). Optimum conditions were: 20 mL ethanol:water 60:40 v/v, 20 min, 100 °C, sample weight 0.1 g for MAE and 20 mL ethanol:water 50:50 v/v, 2 h, 55 °C, sample weight 0.5 g for CE. Root extracts obtained by MAE (the most efficient technique) presented the highest phenolic (47.7 ± 0.9 mg gallic acid equivalents/g dry weight) and flavonoid (17.1 ± 0.8 mg epicatechin equivalents/g dry weight) content, and antioxidant activity (28.4 ± 2.0 mg trolox equivalents/g dry weight and 36.1 ± 2.7 mg ascorbic acid equivalents/g dry weight for DPPH-RSA and FRAP assays, respectively), followed by bark and core wood extracts. HPLC-PDA analysis revealed that phloridzin was the main contributor to the phenolic composition representing 52%–87% of the total amount of phenolic compounds quantified, while phenolic acids represents less than 10%. This study reveals the potential of apple tree wood residues valorization through the recovery of phenolic compounds for food, pharmaceutical and cosmetic applications.

1. Introduction

Polyphenols are one of the main groups of secondary plant metabolites, essentially for their normal growth and defense against infection and injury, and their health benefits have been extensively described (El Gharras, 2009; Ghitescu et al., 2015; Stevanovic et al., 2009). In the last few years, search of inexpensive and renewable sources of polyphenols has been attracting researchers interest. For that reason, the number of publications concerning the extraction of these compounds from biomass has been increasing (Bouras et al., 2015; Ghitescu et al., 2015; Hofmann et al., 2015; Lazar et al., 2016).

Every year, Belgium apple farmers renew 6% of the apple plantation, which reflects in the annual production of 30,000 ton of woods residues (FAOSTAT, 2015). Traditionally, these apple tree residues are used in low added value applications, such as firewood or dispersed (Dedrie et al., 2015; Ghitescu et al., 2015). Still, in last few years, tree materials, such as bark, have been emerging as possible sources of valuable compounds (Table 1) (Ghitescu et al., 2015; Hofmann et al., 2015). To this regard, the recovery of phenolic compounds from these wood wastes is gaining considerable attention, especially ascribable to the antioxidant properties that these compounds exert (Kammerer et al., 2014; Stevanovic et al., 2009).

Abbreviations: AA, ascorbic acid; CE, conventional extraction; CV, coefficient variation; DPPH-RSA, 2,2-diphenyl-1-picrylhydrazyl radical scavenging activity; DW, dry weight; FRAP, ferric reduction activity power; GA, gallic acid; GAE, gallic acid equivalents; HPLC-PDA, high performance liquid chromatography with photodiode array detection; MAE, microwave assisted extraction; RSM, response surface methodology; TE, trolox equivalents; TPC, total phenolic content; TPTZ, 2,4,6-tris(2-pyridyl)-s-triazine; Trolox, 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxilic acid; UPLC-DAD, ultra-performance liquid chromatography–diode array detection

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Table 1
Summary of the published reports on the extraction of phenolic compounds from tree wood residues.

Sample	Extraction technique and conditions	TPC[a]	TPC units	Reference
Birch (*Betula pendula*) and pine (*Pinus sylvestris*) bark varieties	Stirring: 500 mg, 2 × 10 mL of methanol:water 80:20 v/v, 1 min	2.0 ± 0.1–76.0 ± 2.9	mg GAE[b]/g DW[c]	Kähkönen et al. (1999)
Port Orford cedar (*Chamaecyparis lawsoniana* (A. Murr.) Parl.) bark	SE[d]: 100 g, 1 L of methanol	77.7–88.8	mg GAE/g DW	Gao et al. (2007)
Pine (*Pinus rigida* × *taeda* and *Pinus koraiensis*) bark	10 g, 100 mL boiling water, 1 h	111–862	mg CE[e]/g extract	Ku et al. (2007)
Bark of *Phyllanthus emblica* L.	MAE: 3 g, 75% aqueous ethanol, 25 min, 45 °C	19.8	%	Yang et al. (2009)
Oak (*Prunus avium* and *Quercus robur*) wood from different countries	500 mg of sawdust, 30 mL methanol, 30 min, RT[f]	6.95 ± 0.37–101.2 ± 1.2	μg GAE/g oak wood	Alañón et al. (2011)
Pinus radiata bark	Maceration: 20 g, 200 mL acetone:water 70:30 v/v, 40 °C	412 ± 0	mg CE/g bark	Aspé and Fernández (2011)
	MAE[g]: 20 g, 200 mL acetone:water 70:30 v/v, 900 W, 2450 MHz	479 ± 49		
	UAE[h]: 20 g, 200 mL acetone:water 70:30 v/v, 35 KHz, 85 W, RT	388 ± 7		
	SE: 20 g, 200 mL acetone:water 70:30 v/v, 82 °C	622 ± 40		
Pinus radiata bark	100 g, 2 L ethanol:water 3:1 w/w, 120 min, 120 °C	0.55 ± 0.01	g GAE/g extract	Bocalandro et al. (2012)
Bark of *Maclura tinctoria* (L.)	Stirring: acetone:water 70:30 v/v, 30 min, RT + 10 min at 85 °C	43.2 ± 1.2	mg GAE/g	Lamounier et al. (2012)
Quercus (*Q. robur L.*) bark	Maceration: 5 g, 100 mL water, 120 min, 25 °C	3.7 ± 0.6	mg GAE/g bark	Bouras et al. (2015)
	Heat reflux: 5 g, 100 mL water, 120 min, 100 °C	11.7 ± 0.5		
	MAE: 5 g, 100 mL water, 120 min, 100 °C, 400 W	16.50 ± 0.07		
French maritime pine bark (*Pinus pinaster*)	Maceration: 1 g, 9 mL of water + 1% NaOH + 0.25% Na_2SO_3 + 0.25% $NaHSO_3$, 2 h, 70 °C	54.2 ± 19.4	mg GAE/g bark	Chupin et al. (2015)
	MAE: 1 g, 10 mL ethanol:water 80:20 v/v, 3 min, 100 W	28.3 ± 2.9		
	Hydrodistillation: 100 g, 1 L water, 180 min	14.3		Mellouk et al. (2015)
	Solvent free MAE: 100 g, 92.4 min, 803.5 W	139.2		
Oak bark *Quercus robur* L. and *Quercus petraea*	SE: 1 g, 70 mL with 2 successive extractions with water and ethanol, 6 h	5.0 ± 0.3–13.4 ± 0.2	mg GAE/g DW	Dedrie et al. (2015)
Bark of *Solidago canadensis* L:	Stirring: 1 g, 20 mL 50% aqueous ethanol, 25 °C, 30 min	0.601 ± 0.041–1.584 ± 0.020	mg GAE/g	Deng et al. (2015)
	UAE: 5 g, 100 mL 50% aqueous ethanol, 30 min			
	HPE[i]: 1 g, 20 mL 50% aqueous ethanol, 25 °C, 500 MPa, 10 min			
Spruce bark	UAE: 10 g, 100 mL ethanol:water 70:30 v/v, 54 °C, 60 min, 35 kHz, 320 W	13.2	mg GAE/g	Ghitescu et al. (2015)
Beech bark (*Fagus sylvatica* L.)	Stirring: 0.15 g, 15 mL ethanol:water 80:20 v/v, 5 h, RT	48.3 ± 1.2	mg QE[j]/g DW	Hofmann et al. (2015)
	Sonication: 0.15 g, 15 mL ethanol:water 80:20 v/v, 10 min, RT	49.9 ± 1.1		
	MAE: 0.15 g, 15 mL ethanol:water 80:20 v/v, 20 min, 120 °C	65.2 ± 5.6		
Moroccan Acacia mollissima bark	Stirring: 1.0 g, 20 mL methanol:water 80:20 v/v, 2 h, 20 °C	353.6 ± 0.5	mg GAE/g bark	Naima et al. (2015)
	Infusion: 1.0 g, 20 mL methanol:water 80:20 v/v, 2 h, 60 °C	258.4 ± 3.6		
	MAE: 1.0 g, 20 mL methanol:water 80:20 v/v, 5 min, 150 W	441.6 ± 0.3		
	MAE: 1.0 g, 20 mL ethanol, 5 min, 150 W	279.7 ± 8.1		
	MAE: 1.0 g, 20 mL water, 5 min, 150 W	298.8 ± 0.6		
Spruce bark (*Picea abies*)	UAE: 5 g, 50 mL ethanol:water 70:30 v/v, 60 °C, 35 kHz, 320 W	43.1	mg GAE/g	Lazar et al. (2016)

[a] TPC: total phenolic content.
[b] GAE: gallic acid equivalents.
[c] DW: dry weight.
[d] SE: Soxhlet extraction.
[e] CE: catechin equivalents.
[f] RT: room temperature.
[g] MAE: microwave assisted extraction.
[h] UAE: ultrasound assisted extraction.
[i] HPE: high hydrostatic pressure-assisted extraction.
[j] QE: quercetin equivalents.

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