

Electronic Supplementary Material

Novel configurations for a citrus waste based biorefinery: from solventless to simultaneous ultrasound and microwave assisted extraction

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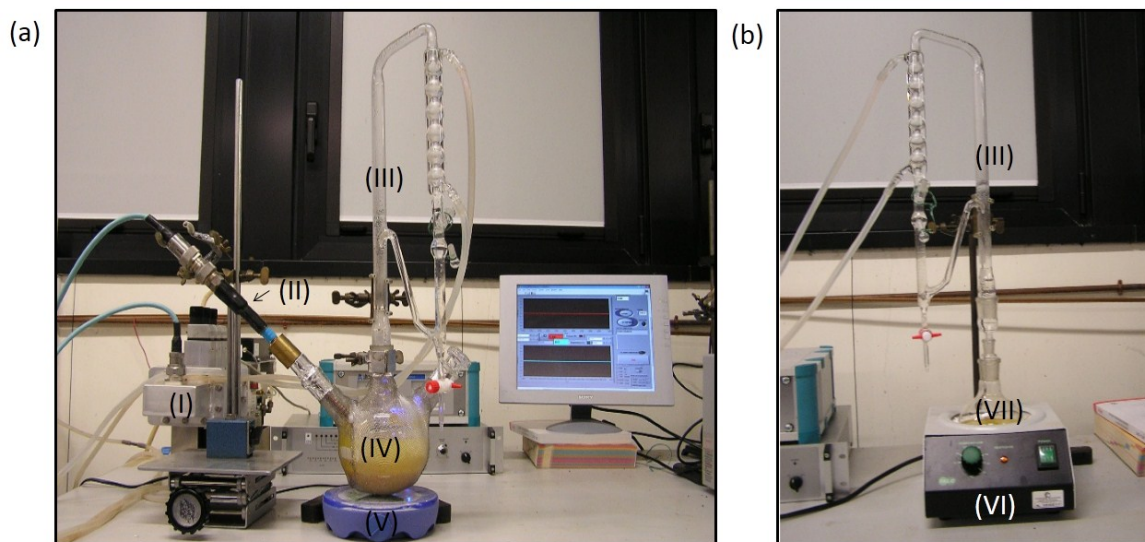


Fig. S1. Experimental extraction configurations used in this work: (a) microwave-assisted hydrodistillation (coaxial MWHD) and, (b) conventional hydrodistillation (CH). Insert details: (I) MW generator, (II) coaxial antenna, (III) clewenger condenser, (IV) 1 L glass vessel, (V) stirrer, (VI) electrical heater, (VII) glass vessel.

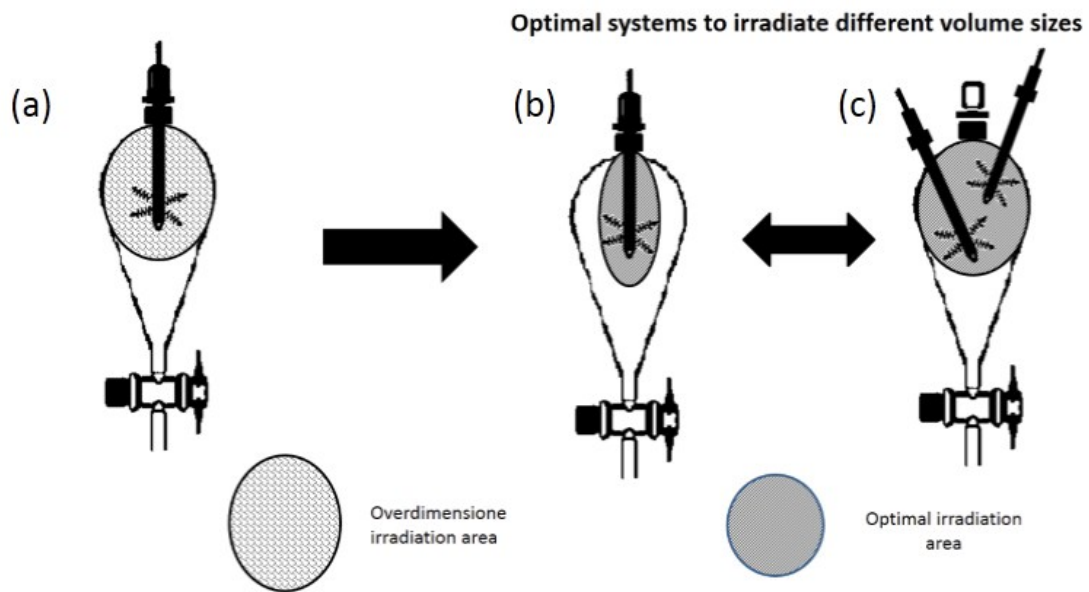


Fig. S2. SMWAE device: (a) oversized holder and, optimal configuration of a solventless MW assisted extractor (b) and (c).

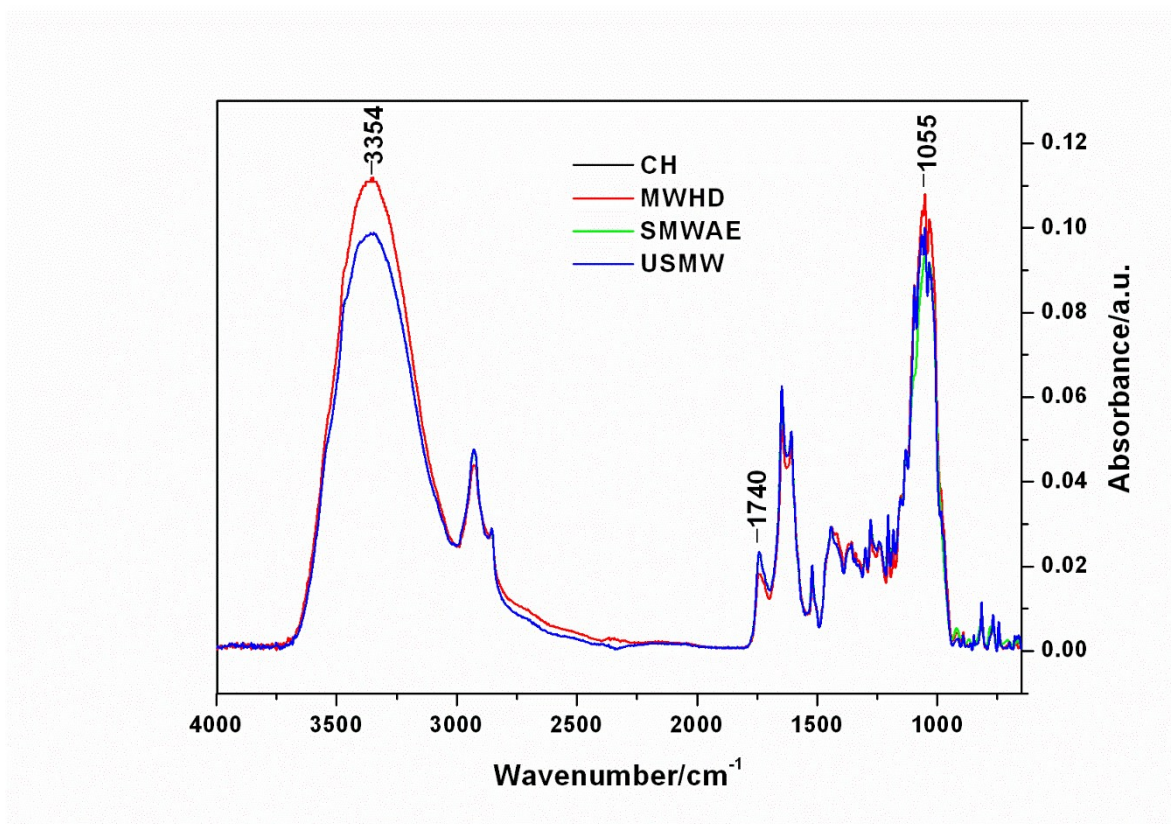


Fig. S3. FTIR spectra of dried orange peel after the EO extraction processes

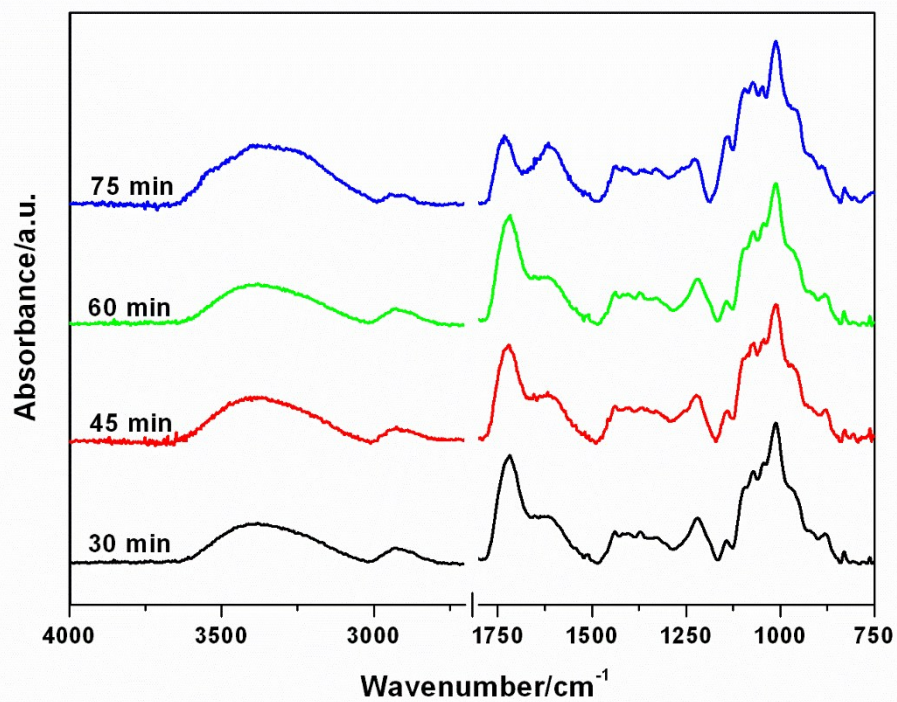


Fig. S4. FTIR spectra of extracted pectin obtained by MWHD using the solid residues produced at different times of EO extraction

Table S1. Chemical composition of the orange peel EO obtained at different extraction times.

No.	Compound	RI	CH			SMWAE		US-MWHD			MWHD		
			t _{extrac}			t _{extrac}		t _{extrac}			t _{extrac}		
			5 min	30 min	120 min	2 min	18 min	5 min	30 min	45 min	5 min	30 min	60 min
Hydrocarbon monoterpenes													
1	α -pinene	937	0.2	0.6	0.8	0.8	0.8	0.6	0.8	0.8	0.3	0.5	0.8
2	sabinene	976	0.2	0.4	0.5	0.5	0.5	0.4	0.5	0.5	0.2	0.3	0.5
3	β -pinene	979	0.1	0.1	0.1	tr	tr	tr	tr	tr	tr	tr	tr
4	myrcene	992	0.7	1.5	2.2	2.2	2.1	1.8	2.2	2.2	0.8	1.4	2.2
5	Δ^3 -carene	1012	0.1	0.1	0.2	0.2	0.2	0.2	0.2	0.2	0.1	0.2	0.2
6	limonene	1034	28.9	66.1	94.4	95.2	95.3	73.2	92.5	95.0	33.0	58.4	94.7
7	γ -terpinene	1061	0.4	0.7	1.0	0.6	0.6	-	-	-	0.3	0.5	0.9
Oxygenated monoterpenes													
8	linalool	1099	0.3	0.5	0.5	0.4	0.3	0.2	0.2	0.2	0.3	0.3	0.5
Hydrocarbon sesquiterpenes													
9	valencene	1491	0.1	0.2	0.3	0.2	0.2	0.2	0.3	0.4	0.1	0.2	0.3
Others													
10	n-decanal	1206	0.1	0.1	0.1	-	-	-	-	-	-	-	-
11	n-octanal	1003	0.1	0.1	0.1	-	-	-	-	-	-	-	-
Yield %			0.48	1.09	1.55	1.16	1.18	1.17	1.49	1.53	0.55	1.29	1.57

Table S2. Pectin isolation from orange peel waste by means of microwave-assisted extraction

Hydrolyzed agent (mM)	Process	Time (min)	T (°C)	Solid:H ₂ O ratio	Ethanol:liquid extracted (v:v) ratio	Yield % (w/w)	reference
HNO ₃ (pH=2)	MW (500W)	3	N.R	1:30	2:1	24	(Boukroufa et al., 2015)
Citric acid (pH=1.5)	MW (700W)	3	N.R.	1:15	N.R.	29	(Hosseini et al., 2016)
H ₂ SO ₄ (pH=1.4)	MW (422W)	2.5	N.R.	1:16.9	1:1	19	(Prakash Maran et al., 2013)
HCl (pH=1.5)	MW (500W)	21	80	1:50	2:1	18	(Guo et al., 2012)
Citric acid (0.5 M)	MW 500W	1.5	100° C	1:20	2:1	16-21%	This work