

Table S1. Calibration curves used in the quantification of polar compounds present in olive oil filter cake.

Commercial standard	Calibration range (mg/L)	Calibration curve	R ²
Quinic acid	0.5–20	$y = 142862x - 99193$	0.9923
Hydroxytyrosol	0.5–20	$y = 33646x - 14770$	0.9851
Oleuropein	0.5–20	$y = 272392x - 80988$	0.9977
Pinoresinol	0.5–20	$y = 47383x - 36828$	0.9853

Table S2. Compositional variations of polyphenols ordered by families for each compound in all extracts, expressed in ug compound/g extract ($X \pm SD$).

Proposed compound	Drying Technique		
	Vacuum-drying	Spray-drying	Freeze-drying
PHENOLIC COMPOUNDS			
Total Phenolic Alcohols	12665 ± 48	10364 ± 243	24066 ± 488
Total Non-oxidized Phenolic Alcohols	6407 ± 289	3705 ± 69	13025 ± 102
Hydroxytyrosol	1548 ± 116	421 ± 15	2620 ± 83
Oxidized hydroxytyrosol	6258 ± 242	6659 ± 215	11041 ± 456
Hydroxytyrosol acetate	4859 ± 179	3284 ± 54	10405 ± 146
Total Secoiridoids	2320 ± 82	2202 ± 22	4654 ± 14
Secoiridoid derivative	753 ± 27	631 ± 2	1012 ± 36
Hydroxy oleuropein aglycon	620 ± 28	532 ± 11	1235 ± 15
Hydroxy decarboxymethyl-ligstroside aglycone	227 ± 11	270 ± 5	770 ± 14
Oleuropein aglycone derivative	412 ± 12	435 ± 19	961 ± 29
Comselogoside	308 ± 7	334 ± 6	676 ± 57
6-O-[(2E)-2,6-Dimethyl-8-hydroxy-2-octenoyloxy] secologanoside	NQ	NQ	NQ
Total Phenolic Alcohols + Secoiridoids	14986 ± 130	12566 ± 265	28720 ± 492
Total Non-oxidized Phenolic Alcohols + Secoiridoids	8727 ± 367	5907 ± 83	17679 ± 93
Total Lignans	1316 ± 39	1131 ± 19	2018 ± 36
(+)-Acetoxypinoresinol	1316 ± 39	1131 ± 19	2018 ± 36
Total Phenolic Compounds	16301 ± 94	13697 ± 248	30738 ± 630
PHENOLIC COMPOUNDS DERIVATIVES (non-phenolic molecules)			
Total oleosides, elenolic acids and derivatives	1601 ± 61	1470 ± 36	3047 ± 11
Hydroxylated product of the dialdehydic form of decarboxymethyl-elenolic acid	NQ	NQ	NQ
Elenolic acid or isomer 1	182 ± 6	156 ± 4	324 ± 7
Elenolic acid or isomer 2	437 ± 18	495 ± 9	961 ± 10

Aldehydic form of decarboxymethyl elenolic acid	148 ± 3	113 ± 1	423 ± 6
Dialdehydic form of decarboxymethyl elenolic acid	NQ	NQ	NQ
Oleoside	833 ± 34	706 ± 27	1338 ± 24
OTHER POLAR COMPOUNDS			
Quinic acid	6357 ± 619	3620 ± 40	2758 ± 212
Total other polar compounds	6357 ± 619	3620 ± 40	2758 ± 212

NQ, not quantitated.

Table S3. Statistical data of the Drying-PLE extraction conditions for phenolic compounds.

Drying system	Hydroxytyrosol	Hydroxytyrosol acetate	Oxidized hydroxytyrosol	Secoiridoid derivative	Hydroxy oleuropein aglycon	Hydroxy decarboxymethyl-ligstroside aglycone	Oleuropein aglycone derivative	Comselogoside
Spray-drying/ Vacuum-drying	1	1	0	1	1	1	0	0
Freeze-drying/ Vacuum-drying	1	1	1	1	1	1	1	1
Freeze-drying/ Spray-drying	1	1	1	1	1	1	1	1
Drying system	Oleoside	Elenolic acid or isomer 1	Dialdehydic form of decarboxymethyl-elenolic acid	Elenolic acid or isomer 2	Total phenolic alcohols	Total secoiridoids	Total lignans	Total phenolic alcohols + secoirid
Spray-drying/ Vacuum-drying	1	1	0	1	1	0	1	1
Freeze-drying/ Vacuum-drying	1	1	1	1	1	1	1	1
Freeze-drying/ Spray-drying	1	1	1	1	1	1	1	1
Drying system	Total non oxidized phenolic alcohols + secoirids	Oleosides + Elenolic acids derivatives	Total phenolic compounds					
Spray-drying/ Vacuum-drying	1	1	1					
Freeze-drying/ Vacuum-drying	1	1	1					
Freeze-drying/ Spray-drying	1	1	1					

1 indicated that the means difference was significant at the 0.05 level.

0 indicated that the means difference was not significant at the 0.05 level.