

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