

Table S1. Metals analysis of Cerasuola-OMWW and fractions performed by ICP-MS

Metal (mg/kg)	Cerasuola-OMWW	PAD428-FR1	PAD428-FR2
Sb, Hg, As, Ag, Cd, Se, Bi, Be	< 0.01	< 0.01	< 0.01
Mo	0.01	0.01	< 0.01
Pb	0.03	0.01	< 0.01
Al	0.30	0.28	0.02
Cr	0.02	0.01	< 0.01
Co	0.02	0.02	< 0.01
Cu	0.60	0.39	0.12
Zn	3.00	2.69	0.07
Fe	1.99	1.52	0.11
Mn	2.17	1.92	0.04
Ni	0.11	0.09	0.01
Na	303.34	274.74	4.10
K	7379.34	6850.37	6.36
Ca	61.74	51.03	2.91
Mg	240.56	216.87	4.05

Method according to Di Mauro et al., (*Food Funct.*, 2017,**8**, 4713-4722)

Table S2. Biophenols in Cerasuola-OMWW and PAD428-FR2 performed by HPLC-DAD analysis

Biophenol (mg/l)	Cerasuola-OMWW	PAD428-FR2*	PAD428-FR2°
Gallic acid	-	-	-
Pyrocatechol	6.59±0.08	5.34±0.07	3.26±0.07
<i>p</i> -Hydroxybenzoic acid	-	-	-
4-Hydroxyphenylacetic acid	traces	traces	traces
Caffeic acid	9.12±0.11	8.55±0.12	5.88±0.08
Floretic acid	21.17±0.21	18.23±0.19	10.61±0.09
Verbascoside	traces	traces	traces
<i>p</i> -Coumaric acid	4.27±0.13	3.28±0.06	traces
<i>Trans</i> -ferulic	-	-	-
Oleuropein	14.32±0.15	12.91±0.12	7.32±0.11

* PAD428-FR2 obtained in laboratory; ° PAD428-FR2 obtained by process scale-up.

Mean values (n=9) ± SD were calculated



Figure S1. Pictures of unabsorbed fraction eluted with water (PAD428-FR1, left) and fraction eluted with water/ethanol (50/50) solution (PAD428-FR2, right).

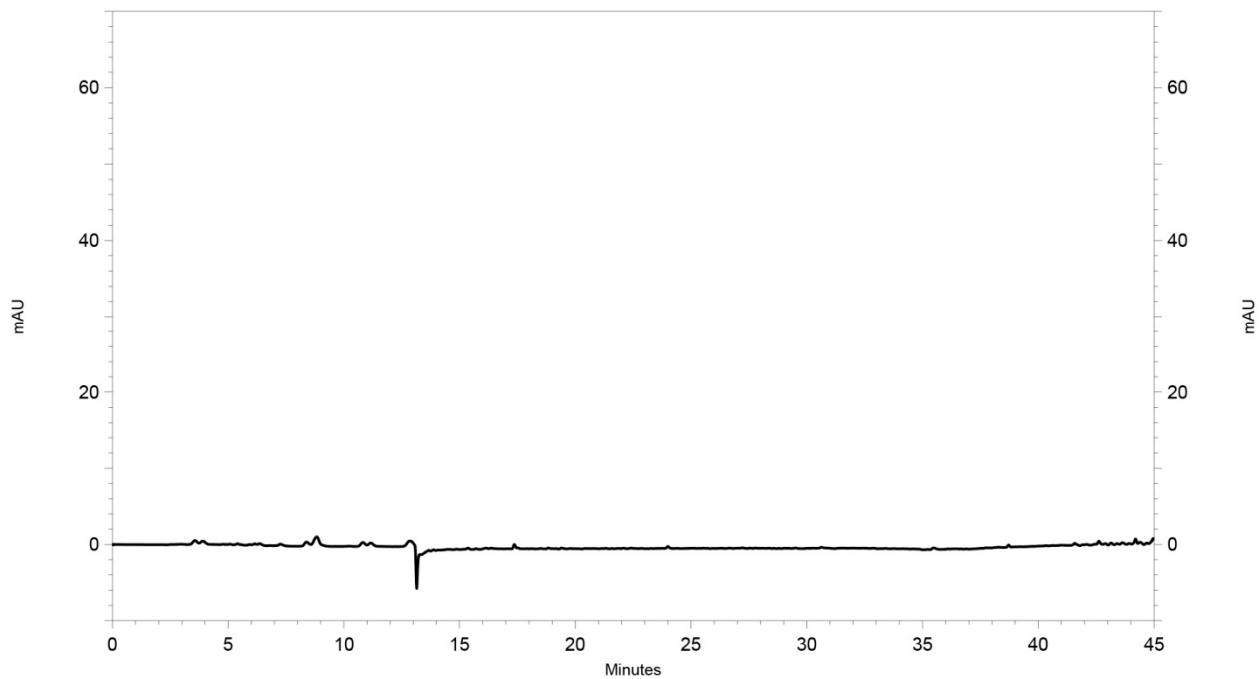


Figure S2. Chromatogram of PAD428-FR1 at 280 nm.

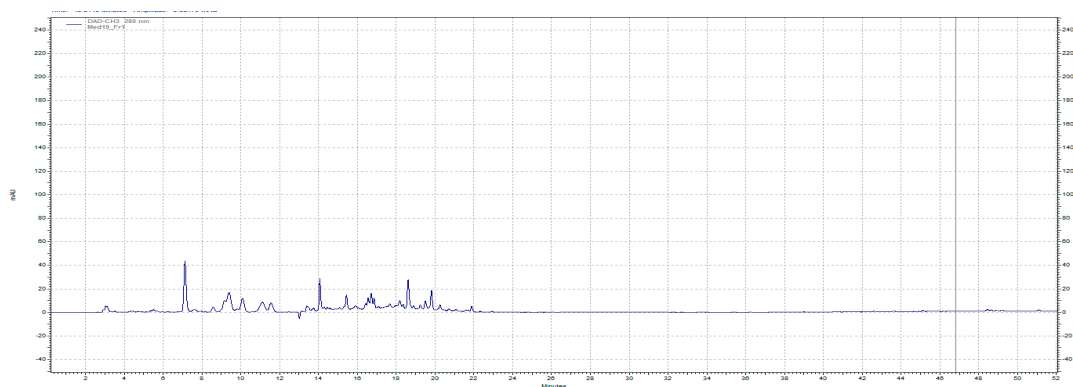


Figure S3. Chromatogram of PAD900-FR1 at 280 nm.

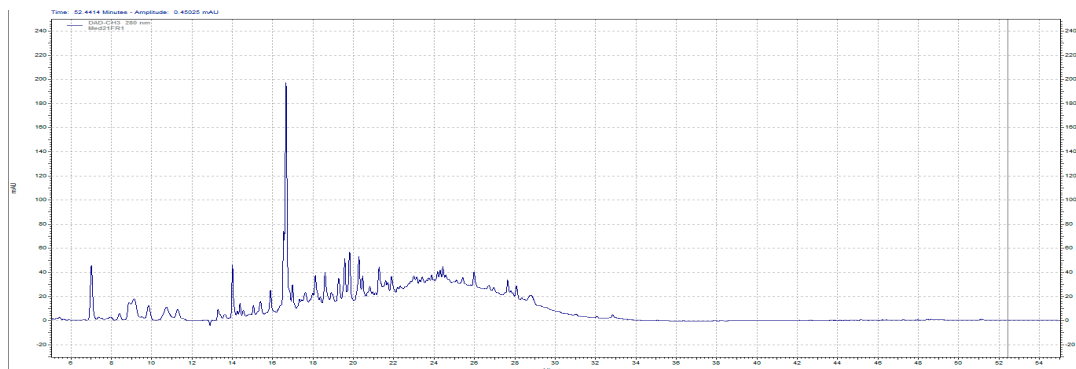


Figure S4. Chromatogram of PAD550-FR1 at 280 nm.

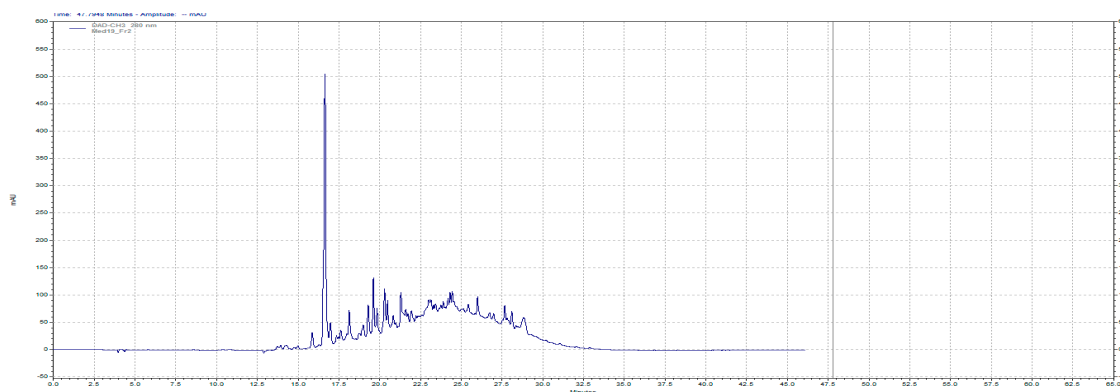


Figure S5. Chromatogram of PAD900-FR2 at 280 nm.

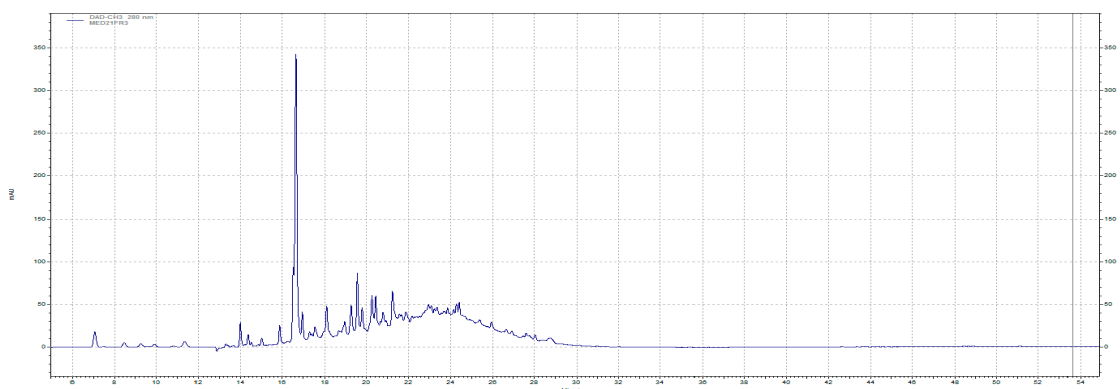


Figure S6. Chromatogram of PAD550-FR2 at 280 nm.

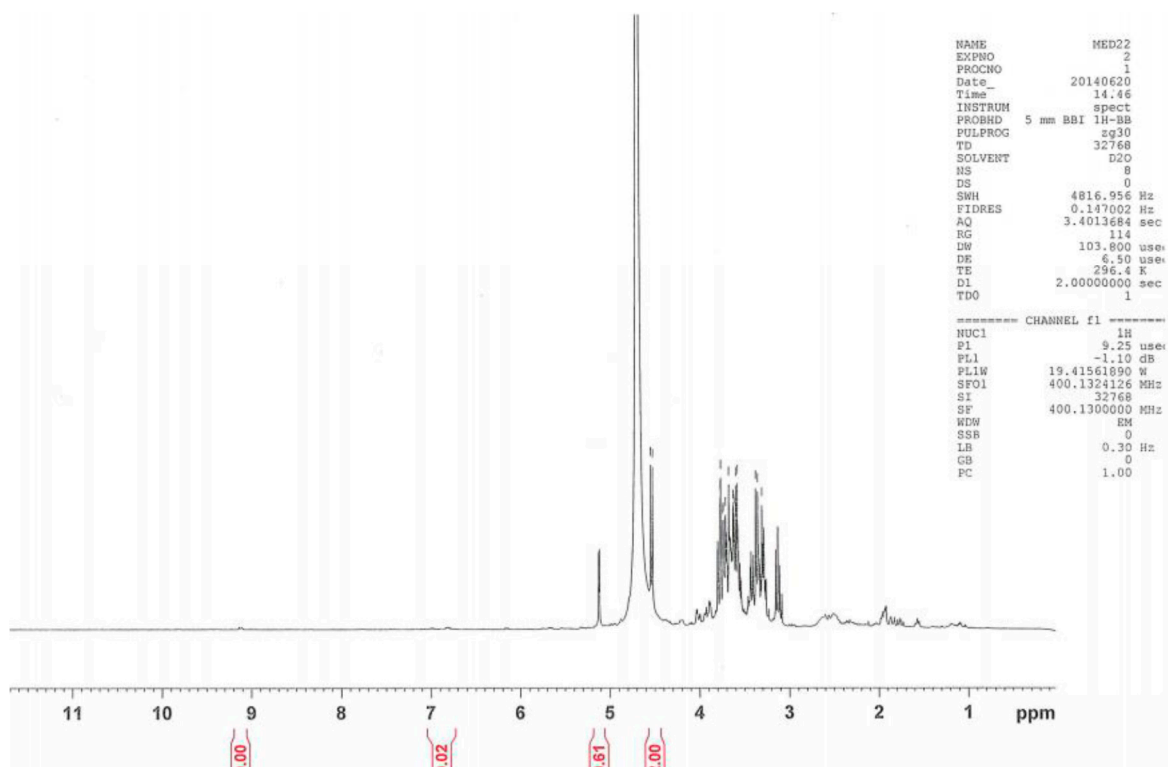


Figure S7. $^1\text{H-NMR}$ spectrum of PAD428-FR1 exhibited only signals in the typical regions of the alkylic (1-2 ppm) and heteroalkylic groups (3-4 ppm), confirming the absence of polyphenols and giving indications about the presence of carbohydrates.

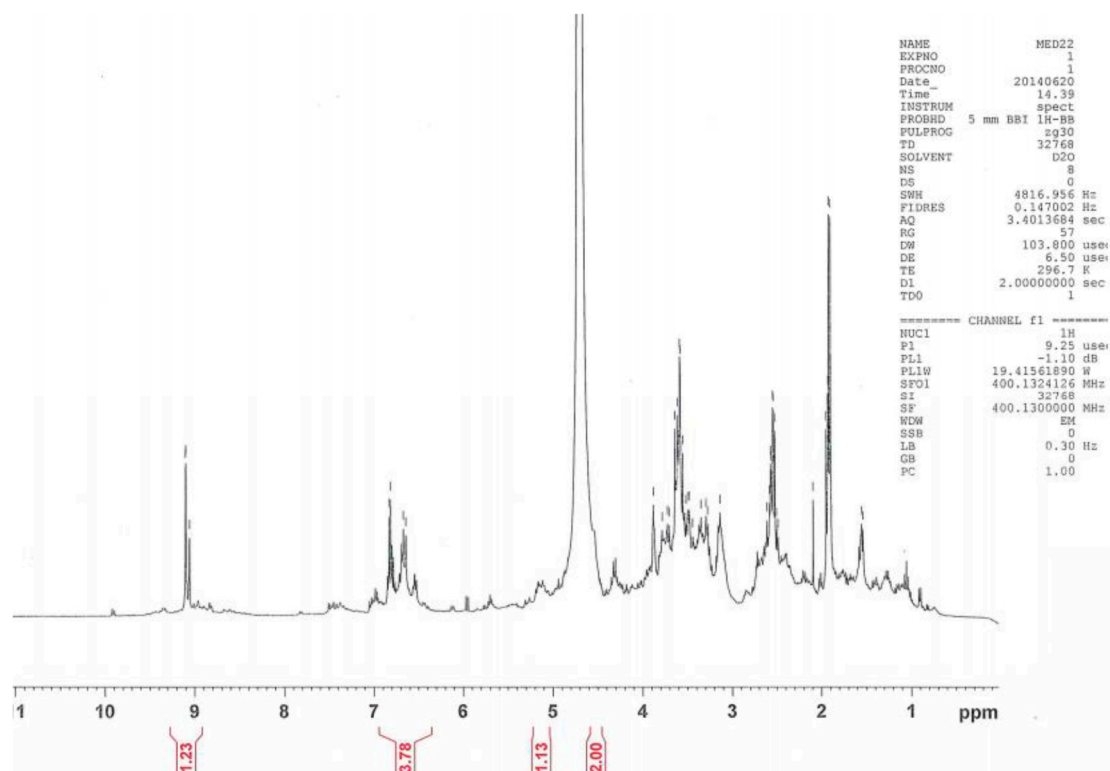


Figure S8. $^1\text{H-NMR}$ spectrum of PAD428-FR2 exhibited the characteristic signals of aromatic compounds (around 6.0-7.5 ppm) confirming the aromatic nature of the compounds in the mixture.

Alkylic (1-2 ppm) and heteroalkylic groups (3-4 ppm) are still present and can be assigned to the sugar moiety of the glycosylated phenols.



Figure S9. Picture of the pilot plant.