Peak	Rt (min)	λmax (nm)	$[M-H]^-$ (*m/z*)	MS^2 (*m/z*)	Tentative identification
1	6.6	279	289	245(199),205(39),179(12)	(+)-Catechin
2	12.3	349	461	315(100)	Isorhamnetin-*O*-deoxyhexoside
3	21.8	348	447	301(100)	Quercetin-*O*-deoxyhexoside
4	24.1	343	431	282(100)	Luteolin-*O*-deoxyhexoside

Table 1: Identification of the four phenolic compounds by HPLC-DAD-ESI/MS

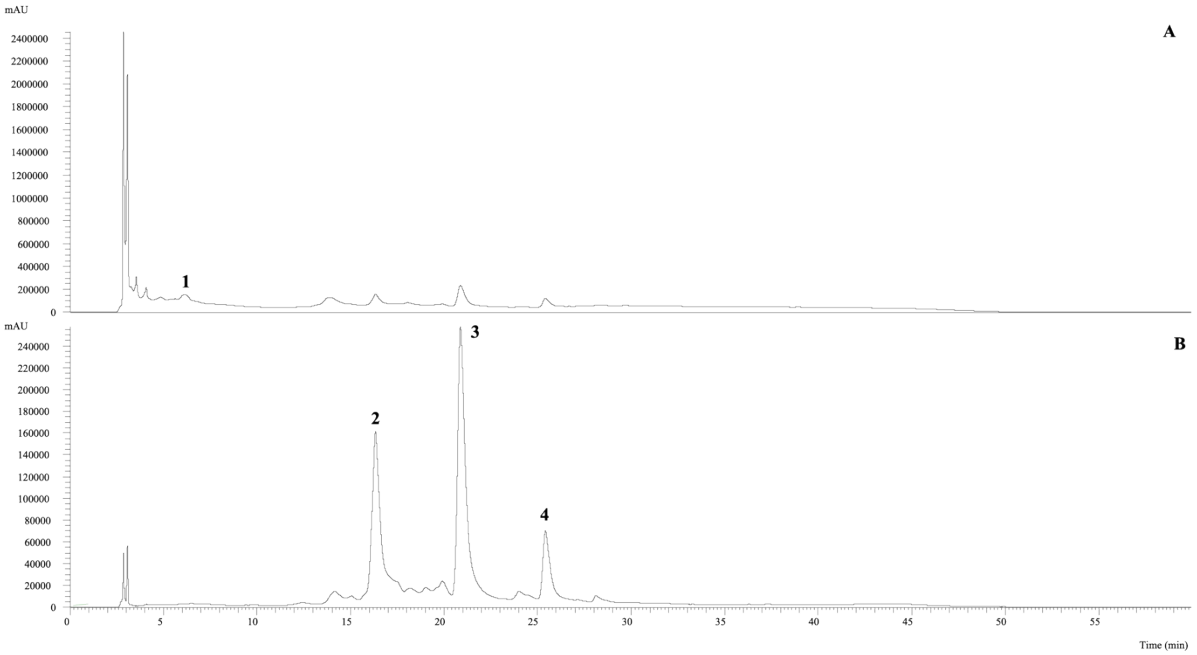


Figure 1: Chromatograms with the identified peaks, from both extractions, A - maceration, B – ultrasound-assisted extraction.

Table 2: Experimental design and responses for DM of *A. unedo* using the Box-Behnken experimental design.

Run	Time (min)	Temperature (ºC)	Solvent (%)	R1 (mg/mL)	R2 (mg/mL)	R3 (mg/mL)	R4 (mg/mL)	R5 (mg/mL)	R6 (mg/mL)
1	10	30	50	10.64	0.331	0.196	0.190	0.141	0.859
2	35	55	50	11.94	3.353	3.032	4.963	1.384	12.732
3	35	55	50	12.46	3.199	3.075	5.228	1.372	13.874
4	35	30	0	8.0	0.641	0.271	0.273	0.156	1.341
5	60	80	50	13.78	2.849	3.816	4.610	1.470	12.744
6	35	55	50	12.98	3.241	3.606	0.870	1.346	13.063
7	35	80	50	9.72	0.351	0.138	0.143	0.000	0.632
8	35	30	100	2.08	1.162	4.019	4.884	1.391	11.457
9	10	55	0	8.10	1.636	1.085	2.173	0.492	5.386
10	60	55	0	3.64	3.374	2.153	2.468	0.981	8.976

11	60	30	50	11.28	1.024	3.468	4.744	1.447	10.685
12	35	80	100	7.90	0.551	0.297	0.843	0.361	2.052
13	35	55	50	12.86	3.857	3.011	4.653	1.817	14.338
14	35	55	50	12.74	3.788	3.556	5.111	1.388	13.843
15	10	55	100	2.52	3.212	3.696	5.505	1.668	14.082
16	60	55	100	8.46	0.356	0.204	0.194	0.145	0.900
17	10	80	50	12.90	2.896	3.705	4.243	1.336	12.180

R1: solid residue; R2: catechin: R3: isorhamnetin; R4: quercetin; R5: luteolin; R6: total phenolic compounds. Highlighted in grey are the center point replicates.

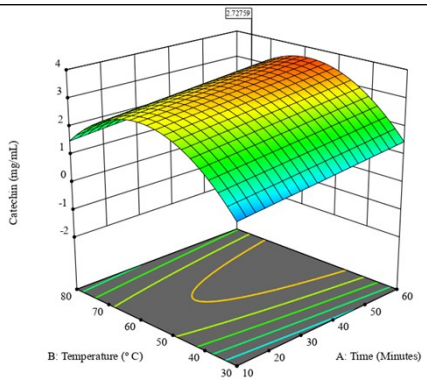
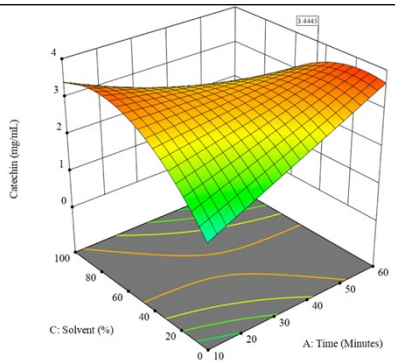
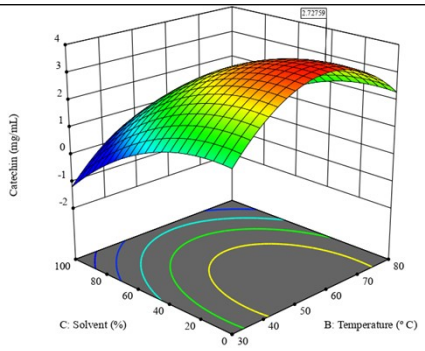
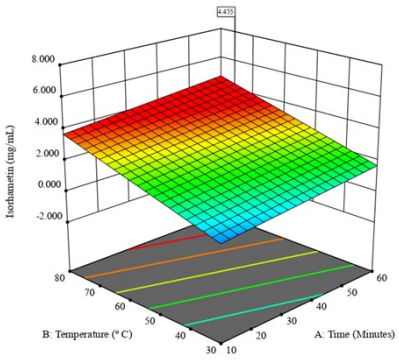
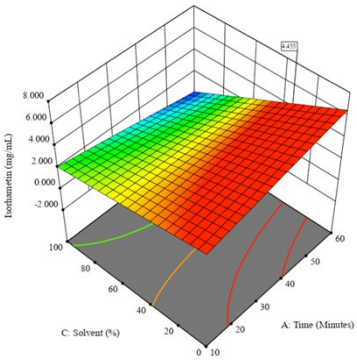
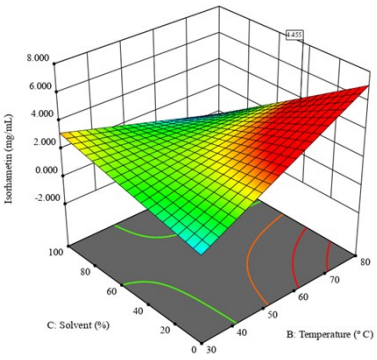
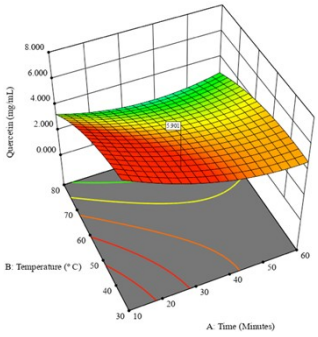
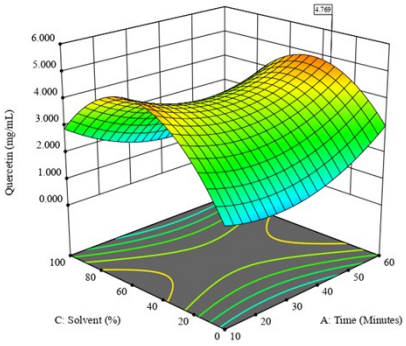
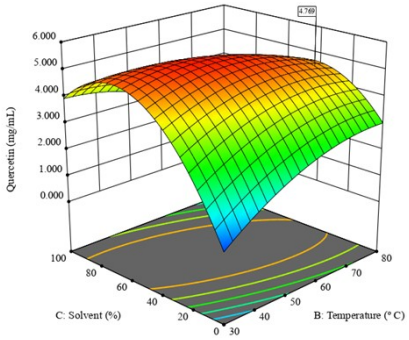
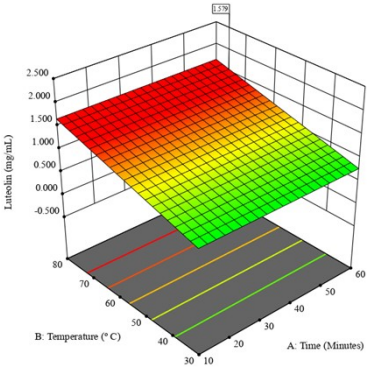
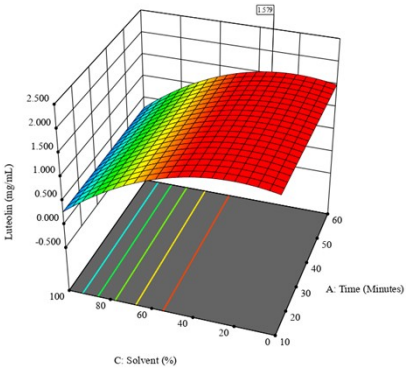
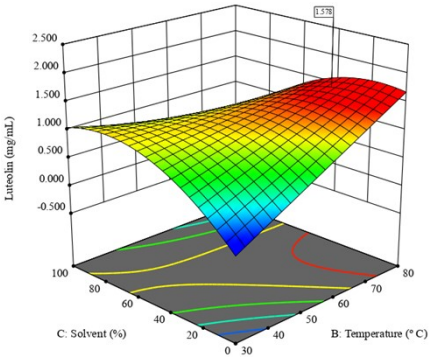
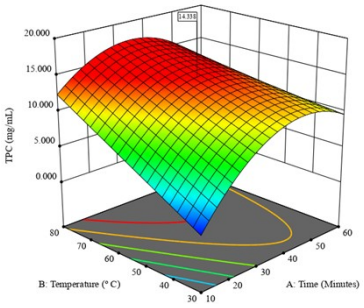
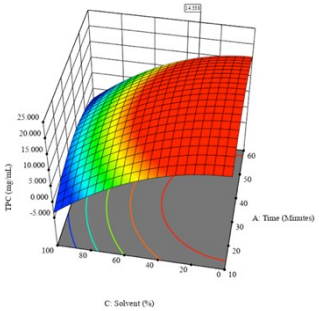
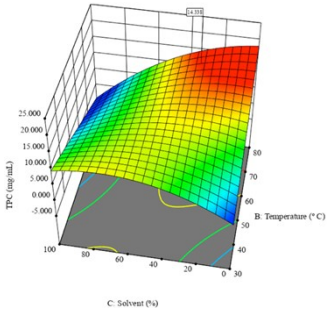
Table 3: Experimental design and responses for UAE of *A. unedo* using the Central Composite Design.

Run	Time (min)	Power (w)	Solvent (%)	R1 (mg/mL)	R2 (mg/mL)	R3 (mg/mL)	R4 (mg/mL)	R5 (mg/mL)	R6 (mg/mL)
1	5	500	50	11.22	1.867	0.490	0.675	0.351	3.384
2	11.25	50	100	0.82	1.961	1.943	2.762	1.097	7.764
3	5	500	100	2.72	0.417	0.179	0.219	0.158	0.975
4	30	387.5	100	1.94	1.434	0.508	0.630	0.242	2.816
5	23.75	162.5	100	0.6	0.433	0.245	0.195	0.143	1.017
6	17.5	275	50	12.06	2.169	1.445	2.273	0.617	6.506
7	23.75	500	25	10.38	3.303	1.978	2.733	0.784	8.799
8	23.75	500	25	9.5	2.362	1.763	2.592	0.824	7.543
9	17.5	500	75	10.62	0.343	0.175	0.207	0.164	0.890
10	17.5	275	50	10.02	0.420	0.210	0.220	0.141	0.993
11	17.5	275	50	13.06	0.352	0.139	0.000	0.000	0.491
12	30	275	0	7.58	0.705	0.607	0.746	0.423	2.482
13	30	50	75	2.64	1.4	0.7	1.000	0.99	4.000
14	17.5	50	0	2.1	2.017	1.421	2.220	0.636	6.296
15	5	387.5	0	9.8	2.910	0.472	1.238	0.464	5.085
16	5	50	0	4.38	1.235	0.309	0.705	0.206	2.456
17	5	50	50	2.7	0.426	0.148	0.150	0.000	0.725
18	17.5	275	50	11.88	1.441	0.834	1.288	0.487	4.052
19	30	50	75	2.72	0.591	0.522	0.507	0.200	1.822
20	17.5	275	0	7.74	0.286	0.138	0.143	0.000	0.567

R1: solid residue; R2: catechin: R3: isorhamnetin; R4: quercetin; R5: luteolin; R6: total phenolic compounds. Highlighted in grey are the center point replicates.

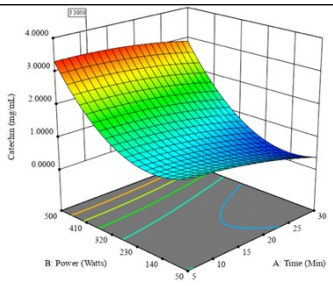
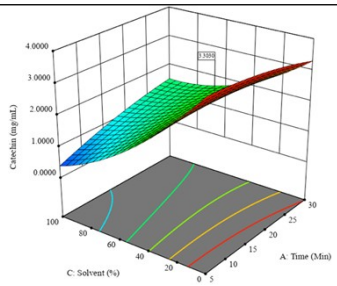
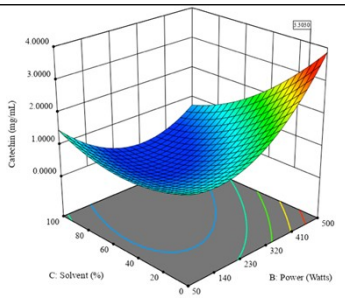
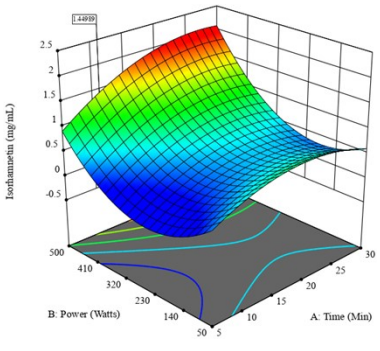
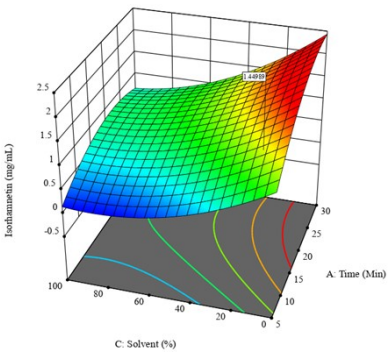
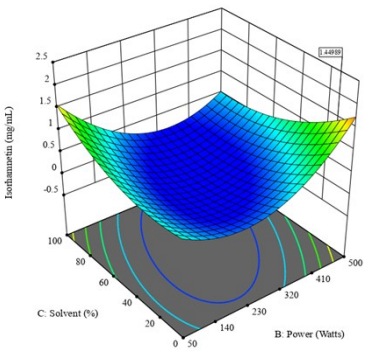
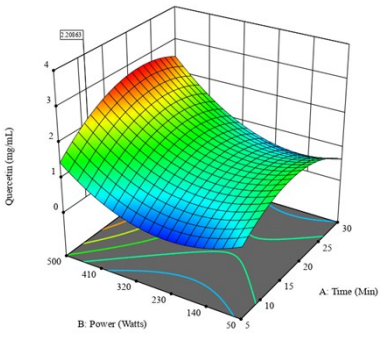
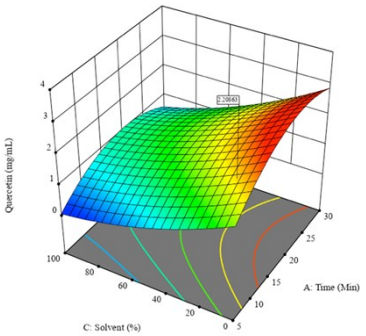
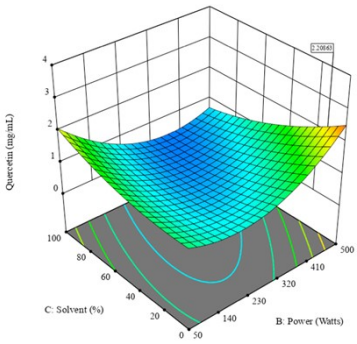
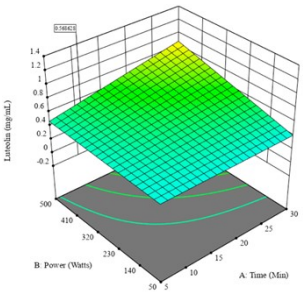
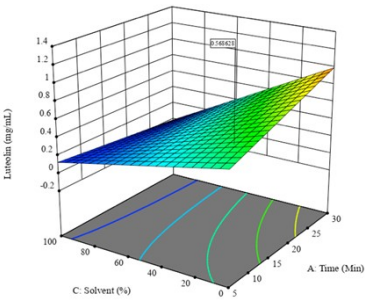
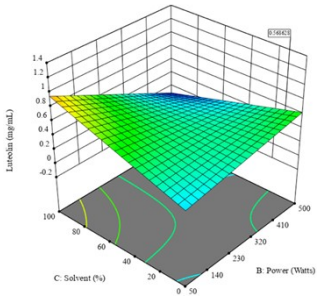
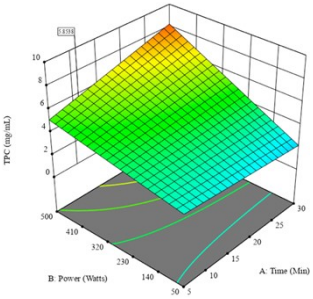
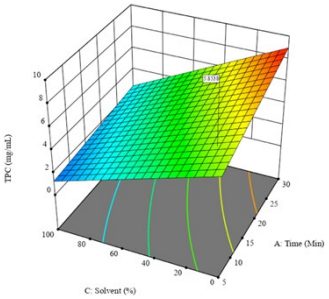
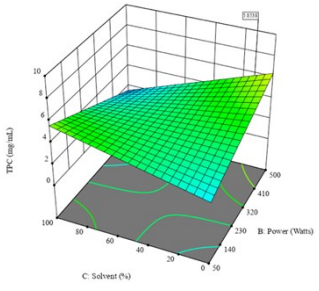
Table 4: Representation of the different response surfaces for the different responses in DM. Each 3D graph shows two of the three factors in the X axis relative to the response (Y axis) when the third response is fixed.

AB – Time vs. Temperature	**AC – Time vs. Solvent**	**BC – Temperature vs. Solvent**

C			
IOD			
QOD			
LOD			
TPC			

Catechin (C), Isorhamnetin-*O*-deoxyhexoside (IOD), Quercetin-*O*-deoxyhexoside (QOD), Luteolin-*O*-deoxyhexoside (LOD), Total phenolic compounds (TPC)

Table 5: Representation of the different response surfaces for the different responses in UAE. Each 3D graph shows two of the three factors in the X axis relative to the response (Y axis) when the third response is fixed.

	AB – Time vs. Power	AC – Time vs. Solvent	BC – Power vs. Solvent
C			
IOD			
QOD			
LOD			
TPC			

Catechin (C), Isorhamnetin-*O*-deoxyhexoside (IOD), Quercetin-*O*-deoxyhexoside (QOD), Luteolin-*O*-deoxyhexoside (LOD), Total phenolic compounds (TPC)