Table S1. Effect of the methanol/water (%) ratio on the phenolic and procyanidins distribution of grape marc extract.

MeOH/ H_2O (%)	Formic acid content (%)	Dry Mass ratio (%)	Sonication time (min)	Shaking time (min)	Total polyphenolic content (mg GAE/g DW)	Catechin (µg/ g DW)	Epicatechin (µg/ g DW)	Procyanidin B1 (µg/ g DW)	Procyanidin C1 (µg/ g DW)
100/ 0	1	100	10	10	6.42 ± 0.61 [a]	76.11 ± 7.02 [b]	19.70 ± 3.76 [a]	ND	28.82 ± 2.40 [a]
80/ 20	1	100	10	10	3.59 ±0.48 [b]	76.98 ± 10.80 [b]	20.10 ± 2.55 [a]	16.73 ± 2.54 [a]	25.96 ± 3.24 [a]
60/ 40	1	100	10	10	5.47 ± 0.29 [a]	98.11 ± 10.37 [a]	26.24 ± 3.38 [a]	21.19 ± 1.77 [a]	37.98 ± 6.43 [a]
40/ 60	1	100	10	10	2.20 ± 0.10 [c]	56.86 ± 10.91 [c]	13.34 ± 3.55 [b]	16.21 ± 2.56 [a]	18.17 ± 3.21 [b]
20/ 80	1	100	10	10	0.80 ± 0.07 [d]	41.20 ± 2.04 [c]	9.68 ± 1.32 [b]	13.32 ± 0.53 [b]	14.32 ± 2.53 [b]
0/ 100	1	100	10	10	0.42 ±0.03 [d]	ND	13.77 ± 0.02 [b]	ND	ND

Results are expressed as mean ± SD, and $p < 0.05$ was considered statistically significant. Statistical significance was calculated by one-way ANOVA followed by Tukey's post-hoc test. Different letters reveal significant differences. "ND" means "not detected".

Table S2. Effect of solid-solvent ratio (%) on the phenolic and procyanidins distribution of grape marc extract.

MeOH/ H_2O (%)	Formic acid content (%)	Sonication time (min)	Shaking time (min)	Solid solvent ratio (%)	Total polyphenolic content (mg GAE/g DW)	Catechin (µg/ g DW)	Epicatechin (µg/ g DW)	Procyanidin B1 (µg/ g DW)	Procyanidin C1 (µg/ g DW)
60/ 40	1	10	10	25	9.55 ± 1.51 [a]	303.27 ± 12.18 [a]	221.60 ± 5.51 [a]	111.80 ± 2.79 [a]	64.36 ± 3.77 [a]
60/ 40	1	10	10	50	9.18 ± 1.61 [a]	319.10 ± 9.12 [a]	226.04 ± 2.92 [a]	96.16 ± 7.97 [b]	45.63 ± 2.96 [a]
60/ 40	1	10	10	75	4.08 ± 0.18 [b]	150.17 ± 3.15 [b]	102.93 ± 7.28 [b]	58.4 ± 3.43 [b]	50.10 ± 4.90 [a]
60/ 40	1	10	10	100	8.10 ± 0.60 [a]	215.00 ± 9.28 [b]	247.56 ± 11.82 [a]	55.70 ± 4.04 [b]	28.60 ± 1.55 [b]
60/ 40	1	10	10	125	5.43 ± 1.16 [b]	112.33 ± 4.01 [b]	195.15 ± 4.61 [a]	36.47 ± 5.42 [c]	21.90 ± 3.96 [b]
60/ 40	1	10	10	150	2.94 ± 0.59 [b]	88.68 ± 8.53 [b]	91.85 ± 8.69 [b]	28.77 ± 2.46 [c]	15.47± 1.37 [b]

Results are expressed as mean ± SD, and $p < 0.05$ was considered statistically significant. Statistical significance was calculated by one-way ANOVA followed by Tukey's post-hoc test. Different letters reveal significant differences. "ND" means "not detected".

Table S3. Effect of the formic acid content (%) on the phenolic and procyanidins distribution of grape marc extract.

MeOH/ H₂O (%)	Formic acid content (%)	Sonication time (min)	Shaking time (min)	Solid solvent ratio (%)	Total polyphenolic content (mg GAE/g DW)	Catechin (μg/ g DW)	Epicatechin (μg/ g DW)	Procyanidin B1 (μg/ g DW)	Procyanidin C1 (μg/ g DW)
60/ 40	0	10	10	100	4.37 ± 1.15 [b]	32.84 ± 3.40 [c]	23.03 ± 1.90 [b]	20.46 ± 1.00 [b]	15.46 ± 1.74 [b]
60/ 40	0.5	10	10	100	4.46 ±0.93 [b]	73.35 ± 3.70 [b]	45.53 ± 3.08 [b]	24.87 ± 0.89 [b]	13.06 ± 0.01 [b]
60/ 40	1	10	10	100	8.10 ± 0.60 [a]	215.00 ± 9.28 [a]	247.56 ± 11.82 [a]	55.70 ± 4.04 [a]	28.60 ± 1.55 [a]
60/ 40	1.5	10	10	100	4.36 ± 0.64 [b]	97.127 ± 5.96 [b]	64.97 ± 0.90 [b]	22.78 ± 0.75 [b]	22.46 ± 4.20 [a]
60/ 40	2	10	10	100	5.15 ± 1.30 [b]	89.89 ± 1.88 [b]	60.26 ± 1.32 [b]	30.69 ± 2.85 [b]	23.50 ± 4.35 [a]

Results are expressed as mean ± SD, and $p < 0.05$ was considered statistically significant. Statistical significance was calculated by one-way ANOVA followed by Tukey's post-hoc test. Different letters reveal significant differences. "ND" means "not detected".

Table S4. Effect of the sonication time (min) on the phenolic and procyanidins distribution of grape marc extract.

MeOH/ H₂O (%)	Formic acid content (%)	Sonication time (min)	Shaking time (min)	Solid solvent ratio (%)	Total polyphenolic content (mg GAE/g DW)	Catechin (μg/ g DW)	Epicatechin (μg/ g DW)	Procyanidin B1 (μg/ g DW)	Procyanidin C1 (μg/ g DW)
60/ 40	1	0	10	100	2.30 ± 0.54 [b]	78.62 ± 2.31 [b]	103.84 ± 2.21 [c]	28.85 ± 2.84 [b]	15.13 ± 2.40 [a]
60/ 40	1	5	10	100	5.36 ± 0.36 [b]	124.03 ± 5.71 [b]	150.86 ± 5.51 [b]	38.74 ± 1.24 [b]	20.32 ± 0.45 [a]
60/ 40	1	10	10	100	8.10 ± 0.60 [a]	215.00 ± 9.28 [a]	247.56 ± 11.82 [a]	55.70 ± 4.04 [a]	28.60 ± 1.55 [a]
60/ 40	1	15	10	100	7.50 ± 1.94 [a]	228.00 ± 9.71 [a]	241.99 ± 9.75 [a]	57.68 ± 7.48 [a]	27.84 ± 4.96 [a]
60/ 40	1	20	10	100	6.27 ± 0.23 [a]	227.72 ± 9.46 [a]	236.45 ± 6.93 [a]	60.18 ± 2.10 [a]	30.71± 1.78 [a]

Results are expressed as mean ± SD, and $p < 0.05$ was considered statistically significant. Statistical significance was calculated by one-way ANOVA followed by Tukey's post-hoc test. Different letters reveal significant differences. "ND" means "not detected".

Table S5. Effect of the shaking time (min) on the phenolic and procyanidins distribution of grape marc extract.

MeOH/ H₂O (%)	Formic acid content (%)	Sonication time (min)	Shaking time (min)	Solid solvent ratio (%)	Total polyphenolic content (mg GAE/g DW)	Catechin (µg/ g DW)	Epicatechin (µg/ g DW)	Procyanidin B1 (µg/ g DW)	Procyanidin C1 (µg/ g DW)
60/ 40	1	10	0	100	4.33 ± 0.03 [b]	86.96 ± 6.60 [b]	48.81 ± 2.47 [b]	29.26 ± 0.56 [b]	23.30 ± 2.18 [a]
60/ 40	1	10	5	100	3.57 ± 0.28 [b]	91.43 ± 7.10 [b]	60.35 ± 2.54 [b]	33.72 ± 2.92 [b]	16.80 ± 2.29 [a]
60/ 40	1	10	10	100	8.10 ± 0.60 [a]	215.00 ± 9.28 [a]	247.56 ± 11.82 [a]	55.70 ± 4.04 [a]	28.60 ± 1.55 [a]
60/ 40	1	10	15	100	9.65 ± 0.89 [a]	187.71 ± 10.86 [a]	150.29 ± 2.84 [a]	53.96 ± 4.59 [a]	24.91 ± 1.24 [a]
60/ 40	1	10	20	100	3.85 ± 1.07 [b]	128.74 ± 2.01[b]	90.36 ± 9.00 [b]	50.08 ± 3.73 [a]	23.96 ± 1.40 [a]

Results are expressed as mean ± SD, and $p < 0.05$ was considered statistically significant. Statistical significance was calculated by one-way ANOVA followed by Tukey's post-hoc test. Different letters reveal significant differences. "ND" means "not detected".

Table S6. Pearson correlation analysis between antioxidant assay and TFC, TPC, antioxidant activity, and procyanidin content of all experimental matrices.

Factor		FOLIN	DPPH	Total flavonoid content	Catechin	Epicatechin	Procyanidin A2	Procyanidin B1	Procyanidin B2	Procyanidin C1
FOLIN	Pearson coefficient	1	0.72	0.91	0.94	0.76	0.75	0.93	0.51	0.56
	p-value	-	0.045	0.002	0.001	0.030	0.032	0.001	0.190	0.150
DPPH	Pearson coefficient	0.72	1	0.93	0.66	0.32	0.32	0.51	0.04	0.30
	p-value	0.045	-	0.001	0.045	0.440	0.440	0.020	0.920	0.470
Total flavonoid	Pearson coefficient	0.91	0.93	1	0.87	0.60	0.50	0.78	0.28	0.42

content										
	p-value	0.002	0.001	-	0.005	0.034	0.040	0.023	0.500	0.300
Catechin	Pearson coefficient	0.94	0.66	0.87	1	0.75	0.75	0.95	0.48	0.57
	p-value	0.001	0.075	0.075	-	0.031	0.032	0.001	0.230	0.140
Epicatechin	Pearson coefficient	0.76	0.32	0.60	0.75	1	1.00	0.83	0.83	0.46
	p-value	0.030	0.440	0.120	0.031	-	0.001	0.011	0.010	0.250
Procyanidin A2	Pearson coefficient	0.75	0.32	0.60	0.75	1.00	1	0.83	0.80	0.41
	p-value	0.032	0.440	0.120	0.032	0.001	-	0.011	0.016	0.320
Procyanidin B1	Pearson coefficient	0.93	0.51	0.78	0.95	0.83	0.83	1	0.53	0.45
	p-value	0.001	0.200	0.023	0.001	0.011	0.011	-	0.180	0.260
Procyanidin B2	Pearson coefficient	0.51	0.04	0.28	0.48	0.83	0.80	0.53	1.00	0.71
	p-value	0.190	0.920	0.500	0.230	0.010	0.016	0.180	-	0.048
Procyanidin C1	Pearson coefficient	0.56	0.30	0.42	0.57	0.46	0.41	0.45	0.71	1
	p-value	0.150	0.470	0.300	0.140	0.250	0.320	0.260	0.048	-