

Selective recovery of glycosylated phenolic compounds from nectarine tree branches (*Prunus persica* var. *nucipersica*)

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Supplementary material

Table A.1: D-optimal design of experiments for the extraction of phenolic compounds from nectarine tree branches.

Exp name	Ethanol concentration (%)	Temperature (°C)
N1	0	40
N2	70	40
N3	100	40
N4	0	65
N5	30	65
N6	100	65
N7	0	90
N8	30	90
N9	70	90
N10	70	65
N11	70	65
N12	70	65

Table A.2: Physical properties of macroporous resins Amberlite XAD-4, XAD-7 and XAD-16.

Resin	XAD-4	XAD-7	XAD-16
Polarity	Non-polar	Moderate polar	Non-polar
Material	Styrenedivinylbenzene	Acrylic ester	Styrenedivinylbenzene
Dry density (g/mL)	1.08	1.24	1.08
Surface area (m ² /g)	725	450	900
Average pore diameter (Å)	50	90	100
Pore volume (mL/g)	0.98	1.14	1.82

Table A.3: Phenolic compounds identified from nectarine tree branches.

Phenolic compound	Retention time (min)	UV detection (nm)	Observed m/z (Da)
Neochlorogenic acid	1.6	320	355.1021 / 163.0387
Catechin	3.0	285	291.0867 / 139.0387
Chlorogenic acid	3.4	320	355.1029 / 163.0390
Procyanidine B2	5.4	285	579.149
Epicatechin	6.1	285	291.0867 / 139.0389
Rutin	10.1	254	611.1616 / 303.0495 / 465.1026
Isoquercetin	10.2	254	465.1035 / 303.0497
Prunin	11.1	285	435.1283 / 273.0754 / 153.0182
Quercetin	14.1	254	303.0509
Naringenin	15.7	285	273.0756
Genistein	15.8	254	271.0598
Apigenin	16.3	320	271.0605
Chrysin	20.4	254	255.0654

Table A.4: Extraction yields obtained during the identification of phenolic compounds from nectarine tree branches.

Phenolic compound	Extraction yield (mg/g _{DM})
Neochlorogenic acid	0.015
Catechin	2.079
Chlorogenic acid	0.918
Procyanidine B2	0.056
Epicatechin	0.068
Rutin	0.363
Isoquercetin	0.144
Prunin	0.187
Quercetin	0.061
Naringenin	0.971
Genistein	0.028
Apigenin	0.014
Chrysin	0.001

Higher extraction yields are highlighted in grey cells.

Table A.5 : Responses values of the design of experiments.

Exp Name	Extraction yield in mg/g _{DM}						G/A ratio
	Catechin	Chlorogenic acid	Naringenin	Rutin	Isoquercetin	Prunin	
N1	1.35	1.18	0.00	0.43	0.14	0.15	0.287
N2	5.87	2.09	0.36	1.06	0.45	0.55	0.248
N3	2.18	0.42	0.32	0.27	0.16	0.37	0.271
N4	2.83	2.00	0.05	0.66	0.21	0.28	0.237
N5	5.68	2.35	0.54	0.88	0.39	0.77	0.238
N6	3.63	0.51	0.72	0.46	0.26	0.47	0.247
N7	3.59	2.05	0.14	0.77	0.24	0.52	0.266
N8	6.41	2.65	0.77	0.97	0.42	1.03	0.247
N9	7.57	3.00	0.84	1.31	0.58	1.07	0.259
N10	6.96	2.73	0.80	1.32	0.56	0.80	0.256
N11	7.84	3.03	0.81	1.42	0.58	0.80	0.240
N12	6.20	2.28	0.68	1.04	0.44	0.82	0.252

Minimum and maximum values are in the grey cells.

Table A.6: Matrix of coefficients and evaluation parameters of the reduced models.

	Coefficients						Evaluation parameters		
	Constant	X ₁	X ₂	X ₁ *X ₁	X ₂ *X ₂	X ₁ *X ₂	R ² adj	p	p lack of fit
Y ₁ - Catechin	7.06	0.59 ^{n.s.}	0.93	-4.03	-	-	0.923	0.000	0.840
Y ₂ - Chlorogenic acid	2.87	-0.41	0.34 ^{n.s.}	-1.66	-	-	0.818	0.001	0.579
Y ₃ - Naringenin	0.69	0.33	0.28	-0.28	-	0.19	0.962	0.000	0.698
Y ₄ - Rutin	1.24	-0.01 ^{n.s.}	0.12 ^{n.s.}	-0.70	-	-	0.709	0.005	0.545
Y ₅ - Isoquercetin	0.52	0.04 ^{n.s.}	0.05 ^{n.s.}	-0.30	-	-	0.844	0.000	0.699
Y ₆ - Prunin	0.86	0.09	0.19	-0.49	0.08	-	0.995	0.000	0.172
Y ₇ - G/A ratio	0.25	-	-0.01 ^{n.s.}	-	0.02	-	0.364	0.053	0.334

X₁: ethanol in water (% (v/v)). X₂: temperature (°C). R² adj: adjusted coefficients of determination. p: p-value of the regression. p lack of fit: p-value for lack of fit of the model. n.s.: non-significant terms.

Table A.7: Validation parameters of different models for the extraction kinetics of the phenolic compounds.

		Peleg's model	1 st order kinetic model	Logarithmic model	2-sites kinetic model
Catechin	R ²	0.9906	0.9918	0.9902	0.9918
	RMSE	0.8008	0.7496	0.8181	0.7496
Chlorogenic acid	R ²	0.9923	0.9938	0.9916	0.9938
	RMSE	0.2570	0.2318	0.2685	0.2318
Naringenin	R ²	0.9972	0.9975	0.9962	0.9975
	RMSE	0.0592	0.0554	0.0689	0.0554
Rutin	R ²	0.9838	0.9853	0.9830	0.9853
	RMSE	0.1706	0.1620	0.1746	0.1620
Isoquercetin	R ²	0.9766	0.9787	0.9760	0.9803
	RMSE	0.0774	0.0740	0.0784	0.0711
Prunin	R ²	0.9991	0.9985	0.9985	0.9989
	RMSE	0.0441	0.0582	0.0573	0.0484

Most satisfying results are in grey cells.

Table A.8: Kinetic and validation parameters of pseudo 1st and pseudo 2nd order models for adsorption.

		Catechin	Chlorogenic acid	Naringenin	Rutin	Isoquercetin	Prunin
Pseudo 1 st order	R ²	0.9842	0.9465	0.9948	0.9943	0.9834	0.9788
	RMSE	3.4040	1.1283	0.4641	0.4219	0.3509	1.1003
	Q _e	26.90	4.75	6.62	6.57	2.83	7.71
	k ₁	0.97	4.02	0.32	0.06	0.22	0.29
Pseudo 2 nd order	R ²	0.9857	0.9465	0.9977	0.9972	0.9916	0.9869
	RMSE	3.2336	1.1283	0.3071	0.2944	0.2487	0.8627
	Q _e	27.87	4.75	6.95	7.51	3.09	8.40
	k ₂	0.05	3923733.71	0.08	0.01	0.09	0.04

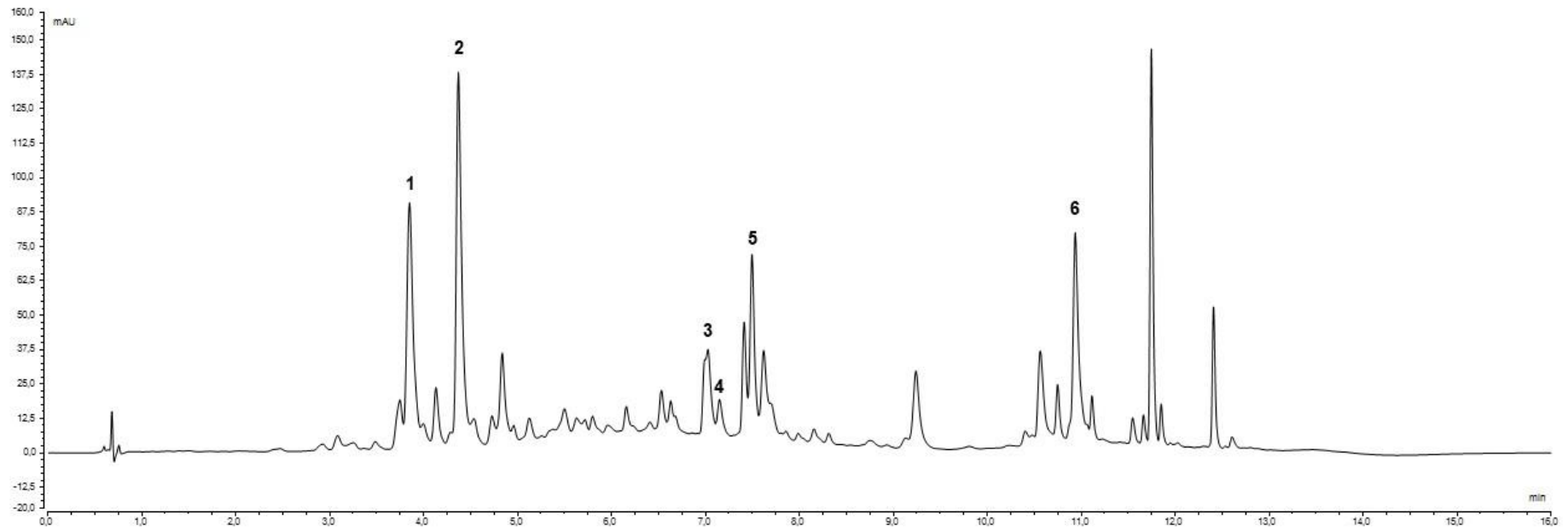


Figure A.1: Chromatogram of the extract at 285 nm. 1: catechin. 2: chlorogenic acid. 3: rutin. 4: isoquercetin. 5: prunin. 6: naringenin.

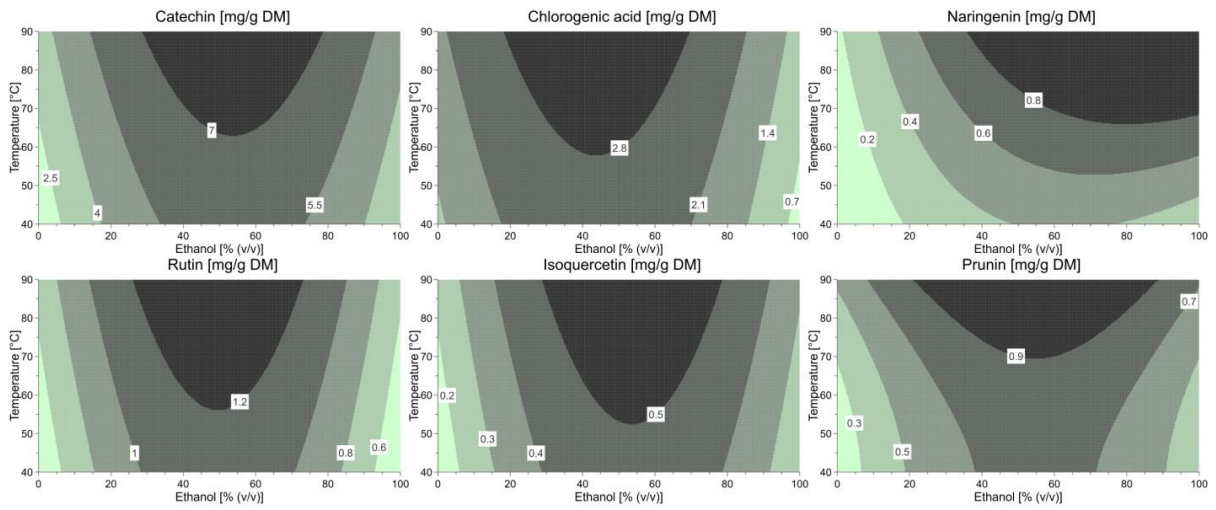


Figure A.2: Surface responses of phenolic compounds from nectarine tree branches.