Potential application of green extracts rich in phenolics for innovative functional foods: Natural deep eutectic solvents as medium for isolation of biocompounds from berries

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Supplementary data

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Table S1. Composition of HBD and HBA that didn't from a eutectic system or formed liquids were too viscosity to use for extractions.

Composition	Molar ratio	Appearance
Choline chloride : Succinic acid	1:1	crystallizes at lower temperature
L–Proline : Succinic acid	1:1	crystallizes at lower temperature
L–Proline : Tartaric acid	1:1	eutectic didn't form
L–Glycine : Lactic acid	1:1	eutectic didn't form
L–Glycine : Glycerol	1:1	eutectic didn't form
L–Glycine : Glycerol	1:3	eutectic didn't form
L–Glycine : Urea	1:1	too viscous
L–Glycine : Succinic acid	1:1	eutectic didn't form
L–Glycine : Citric acid	1:1	too viscous
L–Lysine : Tartaric acid	1:1	eutectic didn't form
L–Lysine : Citric acid	1:1	too viscous
L–Lysine : Succinic acid	1:1	eutectic didn't form
L–Lysine : Glycerol	1:1	eutectic didn't form
L–Lysine : Urea	1:1	eutectic didn't form
L–Lysine : Glycerol	1:3	crystallizes at lower temperature
L–Tyrosine : Lactic acid	1:3	eutectic didn't form
L–Tyrosine : Glycerol	1:1	eutectic didn't form
L–Tyrosine : Glycerol	1:3	eutectic didn't form
L–Tyrosine : Malic acid	1:1	eutectic didn't form
L–Tyrosine : Lactic acid	1:1	eutectic didn't form

Table S2. Optimization of mobile phase for phenolic profile of various berry fruits.

Mobile phase	Composition	Comment	Reference
MP1	ethyl acetate: methanol: acetic acid: formic acid: water 27/2/2/2/2 *v/v/v/v/v*	immiscible mobile phase	26
MP2	ethyl acetate: 2- butanone: formic acid: water 7/3/1.2/0.8 *v/v/v/v*	good separation, all zones are at lower R_F, tailing of zones	27
MP3	ethyl acetate: 2- butanone: formic acid: water 5/3/2/1 *v/v/v/v*	good separation, anthocyanins at lower R_F, other phenolics at higher R_F, tailing of zones	28
MP4	ethyl acetate: water: acetic acid: formic acid 100/27/11/11 *v/v/v/v*	good separation, anthocyanin at lower R_F, other phenolics at higher R_F, sharp and well – defined zones	29
MP5	ethyl acetate: acetic acid: formic acid: water 5/2/2/1 *v/v/v/v*	tailing of zones, appearance of secondary front	modification of MP4
MP6	ethyl acetate: water: acetic acid: formic acid 100/35/11/11 *v/v/v/v*	appearance of secondary front	modification of MP4
MP7	ethyl acetate: water: formic acid 100/30/20 v/v/v	appearance of secondary front	30
MP8	toluene: ethyl acetate: formic acid 5/4/1 *v/v/v*	good separation, anthocyanins are at starting zone, other phenolics are at higher R_F	31
MP9	ethyl acetate: 2- butanone: formic acid: acetic acid: water 5/3/2/2/1 *v/v/v/v/v*	blurry zones	modification of MP2

Table S3. Optimization of mobile phase for metabolic profile of various berry fruits.

Mobile phase	Composition	Comment	Reference
MP1	ethyl acetate: water: acetic acid: formic acid 100/27/11/11 *v/v/v/v*	too polar, zones at high R_F, together with solvent front	29
MP2	ethyl acetate: 2- butanone: formic acid: water 7/3/1.2/0.8 *v/v/v/v*	too polar, zones at high RF, together with solvent front	40,41
MP3	ethyl acetate: toluene: formic acid: water 10/3/1.2/0.8 *v/v/v/v*	immiscible mobile phase	40,41
MP4	ethyl acetate: 2: butanone: formic acid: water 7/3/1/1.5 *v/v/v/v*	stationary phase silica gel NH2, zones didn't appear	42
MP5	ethyl acetate: toluene: formic acid: water 16/4/3/2 *v/v/v/v*	too polar, zones at higher R_F	43
MP6	toluene: ethyl acetate 7/3 *v/v*	too polar, zones at higher R_F	–
MP7	toluene: ethyl acetate 6/4 *v/v*	good separation, blurry zones	–
MP8	toluene: ethyl acetate: formic acid 6/4/1 *v/v/v*	good separation, sharp and well- defined zones	–

Figure S1. HPTLC profiles of methanolic extracts and phenolic standards. Abbreviations: A-chokeberry K1, B- blueberry K1, C- black goji berry K1, CGA- chlorogenic acid, GA- gallic acid, RUT- rutin.

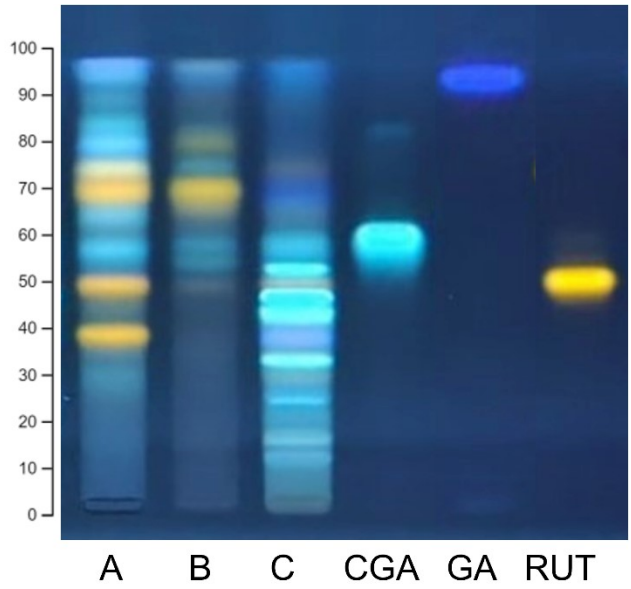

Figure S2. Principal component analysis of NADES extracts for HPTLC phenolic profiles loading plot for A) chokeberry; B) blueberry; C) black goji berry.

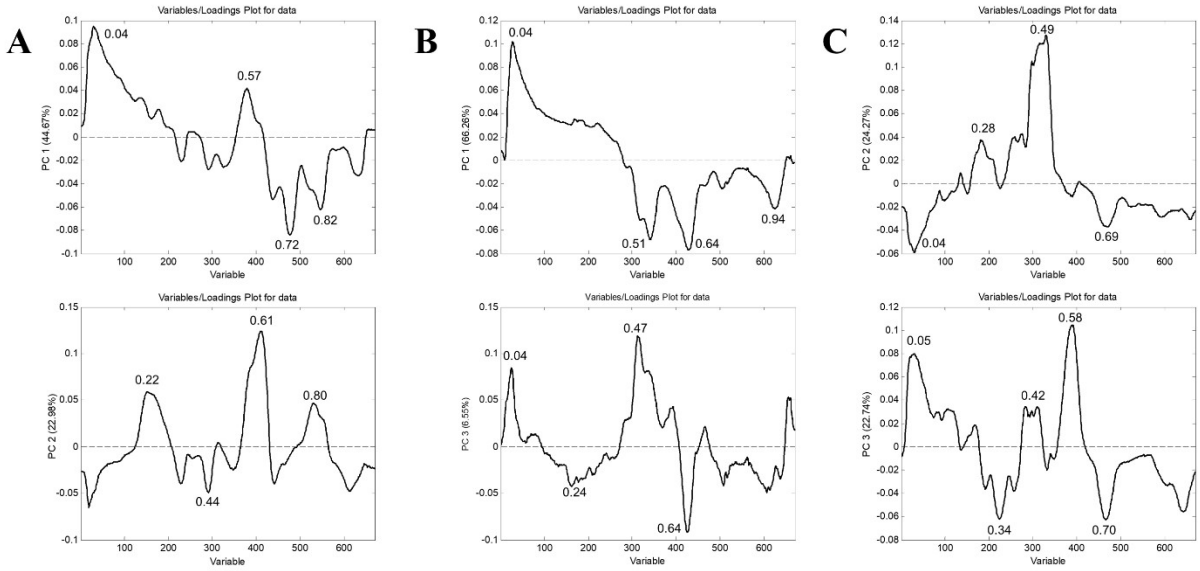

Table S4. Total phenolic content (TPC), total flavonoid content (TFC), total anthocyanin content (TAC) and radical scavenging activity (RSA) in NADES and conventional extracts (mean value for duplicate ± standard deviation).

Chokeberry				
NADES	**TPC (g GAE/kg)**	**TFC (g QUE/kg)**	**RSA (mol TE/kg)**	**TAC (g cyd-3-glu/kg)**
E1	20.6 ± 1.5	43.3 ± 5.8	129.2 ± 8.2	1.21 ± 0.31
E2	23.6 ± 1.7	42.2 ± 5.6	111.0 ± 7.0	0.20 ± 0.05
E3	15.5 ± 1.1	26.8 ± 3.6	87.4 ± 5.5	0.15 ± 0.04
E4	21.0 ± 1.5	40.0 ± 5.3	135.4 ± 8.6	0.13 ± 0.03
E5	29.6 ± 2.2	64.2 ± 8.5	137.1 ± 8.7	4.84 ± 1.25
E6	21.8 ± 1.6	42.9 ± 5.7	124.9 ± 7.9	0.28 ± 0.07
E7	24.9 ± 1.8	50.5 ± 6.7	138.6 ± 8.8	0.30 ± 0.08
E8	14.0 ± 1.0	26.5 ± 3.5	70.6 ± 4.5	1.37 ± 0.35
E9	10.0 ± 0.7	14.6 ± 1.9	142.8 ± 9.0	0.08 ± 0.02
E10	12.2 ± 0.9	20.7 ± 2.8	64.3 ± 4.1	0.12 ± 0.03
E11	33.6 ± 2.4	48.4 ± 6.4	150.9 ± 9.5	2.94 ± 0.76
E12	30.5 ± 2.2	45.4 ± 6.0	163 ± 11	4.07 ± 1.05
E13	26.4 ± 1.9	36.2 ± 4.8	148.8 ± 9.4	3.83 ± 0.99
E14	28.1 ± 2.0	59.0 ± 7.8	156 ± 10	3.51 ± 0.91
E15	27.6 ± 2.0	64.8 ± 8.6	147.2 ± 9.3	0.53 ± 0.14
E16	29.3 ± 2.1	53.4 ± 7.1	147.9 ± 9.3	0.29 ± 0.07
E17	29.6 ± 2.2	52.6 ± 7.0	150.0 ± 9.5	0.32 ± 0.08
E18	29.0 ± 2.1	52.9 ± 7.0	138.1 ± 8.7	0.61 ± 0.16
E19	24.3 ± 1.8	44.6 ± 5.9	124.7 ± 7.9	1.78 ± 0.46
E20	12.2 ± 0.9	16.9 ± 2.2	54.9 ± 3.5	0.09 ± 0.02
E21	34.9 ± 2.5	49.9 ± 6.6	138.9 ± 8.8	0.73 ± 0.19
E22	8.2 ± 0.6	26.5 ± 3.5	48.1 ± 3.0	0.04 ± 0.01
E23	12.7 ± 0.9	19.2 ± 2.5	63.8 ± 4.0	0.05 ± 0.01
E24	15.9 ± 1.2	24.3 ± 3.2	107.3 ± 6.8	0.11 ± 0.03
E25	32.6 ± 2.4	43.8 ± 5.8	145.9 ± 9.2	0.25 ± 0.06
E26	18.3 ± 1.3	27.1 ± 3.6	117.0 ± 7.4	1.60 ± 0.41
E27	14.9 ± 1.1	20.0 ± 2.7	109.6 ± 6.9	1.16 ± 0.30
E28	14.6 ± 1.1	0.13 ± 0.02	33.9 ± 2.1	0.49 ± 0.13
E29	14.7 ± 1.1	7.5 ± 1.0	48.1 ± 3.0	1.25 ± 0.32
E30	8.1 ± 0.6	20.0 ± 2.7	55.7 ± 3.5	0.08 ± 0.02
E31	11.0 ± 0.8	18.3 ± 2.4	55.0 ± 3.5	0.13 ± 0.03
E32	11.3 ± 0.8	18.1 ± 2.4	59.3 ± 3.7	0.09 ± 0.02
E33	14.8 ± 1.1	21.3 ± 2.8	124.1 ± 7.8	0.16 ± 0.04
E34	14.7 ± 1.1	22.2 ± 2.9	111.0 ± 7.0	0.13 ± 0.03
E35	12.2 ± 0.9	22.5 ± 3.0	62.7 ± 4.0	0.11 ± 0.03
E36	15.3 ± 1.1	8.3 ± 1.1	47.0 ± 3.0	0.02 ± 0.01
K1	19.2 ± 1.4	26.4 ± 3.5	115.9 ± 7.3	0.10 ± 0.02
K2	30.0 ± 2.2	54.2 ± 7.2	158 ± 10	0.20 ± 0.05

Table S4. continued

NADES	Blueberry			
	TPC (g GAE/kg)	TFC (g QUE/kg)	RSA (mol TE/kg)	TAC (g cyd-3-glu/kg)
E1	8.5 ± 0.6	16.7 ± 2.2	40.1 ± 2.5	0.39 ± 0.10
E2	12.6 ± 0.9	10.0 ± 1.3	55.0 ± 3.5	0.12 ± 0.03
E3	9.0 ± 0.7	12.3 ± 1.6	52.6 ± 3.3	0.12 ± 0.03
E4	10.6 ± 0.8	7.6 ± 1.0	44.8 ± 2.8	0.07 ± 0.02
E5	14.3 ± 1.0	26.3 ± 3.5	69.0 ± 4.4	2.63 ± 0.68
E6	10.7 ± 0.8	13.2 ± 1.8	54.0 ± 3.4	0.18 ± 0.05
E7	12.7 ± 0.9	21.5 ± 2.9	64.5 ± 4.1	0.18 ± 0.05
E8	9.8 ± 0.7	11.8 ± 1.6	47.9 ± 3.0	1.05 ± 0.27
E9	9.7 ± 0.7	7.1 ± 0.9	49.1 ± 3.1	0.09 ± 0.02
E10	9.1 ± 0.7	3.6 ± 0.5	42.0 ± 2.7	0.09 ± 0.02
E11	14.4 ± 1.1	25.5 ± 3.4	57.9 ± 3.7	1.56 ± 0.40
E12	16.8 ± 1.2	16.7 ± 2.2	70.9 ± 4.5	1.86 ± 0.48
E13	15.3 ± 1.1	11.0 ± 1.5	66.3 ± 4.2	2.09 ± 0.54
E14	15.3 ± 1.1	24.7 ± 3.3	68.1 ± 4.3	1.17 ± 0.30
E15	12.8 ± 0.9	22.2 ± 2.9	56.2 ± 3.6	0.28 ± 0.07
E16	16.5 ± 1.2	16.4 ± 2.2	62.8 ± 4.0	0.19 ± 0.05
E17	17.2 ± 1.3	18.0 ± 2.4	57.3 ± 3.6	0.25 ± 0.06
E18	16.2 ± 1.2	15.1 ± 2.0	56.3 ± 3.6	0.35 ± 0.09
E19	9.4 ± 0.7	5.6 ± 0.8	39.8 ± 2.5	0.42 ± 0.11
E20	11.7 ± 0.9	6.7 ± 0.9	45.2 ± 2.9	0.31 ± 0.08
E21	13.7 ± 1.0	6.9 ± 0.9	38.2 ± 2.4	0.39 ± 0.10
E22	6.5 ± 0.5	2.2 ± 0.3	44.5 ± 2.8	0.05 ± 0.01
E23	7.5 ± 0.5	6.3 ± 0.8	36.4 ± 2.3	0.07 ± 0.02
E24	4.0 ± 0.3	1.9 ± 0.3	15.6 ± 1.0	0.02 ± 0.01
E25	14.1 ± 1.0	23.8 ± 3.2	49.7 ± 3.1	0.87 ± 0.22
E26	9.4 ± 0.7	8.6 ± 1.1	42.4 ± 2.7	0.86 ± 0.22
E27	8.6 ± 0.6	15.8 ± 2.1	38.7 ± 2.4	0.85 ± 0.22
E28	12.1 ± 0.9	8.4 ± 1.1	31.5 ± 2.0	0.30 ± 0.08
E29	11.7 ± 0.9	3.0 ± 0.4	33.7 ± 2.1	0.76 ± 0.20
E30	3.2 ± 0.2	2.1 ± 0.3	21.7 ± 1.4	0.03 ± 0.01
E31	7.1 ± 0.5	4.5 ± 0.6	41.6 ± 2.6	0.09 ± 0.02
E32	2.8 ± 0.2	11.5 ± 1.5	19.4 ± 1.2	0.03 ± 0.01
E33	7.1 ± 0.5	10.2 ± 1.4	31.7 ± 2.0	0.14 ± 0.04
E34	10.4 ± 0.8	9.7 ± 1.3	46.0 ± 2.9	0.22 ± 0.06
E35	7.9 ± 0.6	7.7 ± 1.0	42.9 ± 2.7	0.10 ± 0.03
E36	12.0 ± 0.9	11.0 ± 1.5	40.0 ± 2.5	0.07 ± 0.02
K1	9.7 ± 0.7	9.7 ± 1.3	48.2 ± 3.0	0.15 ± 0.04
K2	10.5 ± 0.8	7.8 ± 1.0	44.9 ± 2.8	0.08 ± 0.02

Table S4. continued

NADES	Black goji			
	TPC (g GAE/kg)	TFC (g QUE/kg)	RSA (mol TE/kg)	TAC (g cyd-3-glu/kg)
E1	23.1 ± 1.7	10.3 ± 1.4	90.4 ± 5.7	2.17 ± 0.56
E2	27.3 ± 2.0	14.2 ± 1.9	98.7 ± 6.2	0.63 ± 0.16
E3	21.3 ± 1.5	7.7 ± 1.0	113.9 ± 7.2	1.53 ± 0.39
E4	27.7 ± 2.0	15.1 ± 2.0	112.8 ± 7.1	0.53 ± 0.14
E5	33.8 ± 2.5	37.2 ± 4.9	143.1 ± 9.0	4.98 ± 1.28
E6	22.6 ± 1.6	7.3 ± 1.0	108.1 ± 6.8	1.64 ± 0.42
E7	21.7 ± 1.6	8.2 ± 1.1	87.1 ± 5.5	0.92 ± 0.24
E8	24.7 ± 1.8	17.5 ± 2.3	98.9 ± 6.2	3.99 ± 1.03
E9	34.4 ± 2.5	2.4 ± 0.3	83.2 ± 5.3	0.67 ± 0.17
E10	18.2 ± 1.3	20.8 ± 2.8	48.6 ± 3.1	0.67 ± 0.17
E11	41.8 ± 3.0	45.4 ± 6.0	133.0 ± 8.4	4.80 ± 1.24
E12	35.7 ± 2.6	34.7 ± 4.6	128.3 ± 8.1	4.20 ± 1.08
E13	34.0 ± 2.5	23.8 ± 3.2	126.2 ± 8.0	3.56 ± 0.92
E14	35.0 ± 2.6	32.0 ± 4.3	119.6 ± 7.6	4.53 ± 1.17
E15	33.6 ± 2.4	26.8 ± 3.6	113.3 ± 7.2	4.88 ± 1.26
E16	37.3 ± 2.7	30.3 ± 4.0	126.9 ± 8.0	2.89 ± 0.75
E17	37.9 ± 2.8	30.3 ± 4.0	132.6 ± 8.4	2.03 ± 0.52
E18	33.0 ± 2.4	20.1 ± 2.7	123.8 ± 7.8	1.43 ± 0.37
E19	21.5 ± 1.6	4.4 ± 0.6	125.9 ± 8.0	2.06 ± 0.53
E20	34.1 ± 2.5	18.9 ± 2.5	115.9 ± 7.3	2.65 ± 0.68
E21	40.0 ± 2.9	27.5 ± 3.7	128.6 ± 8.1	3.17 ± 0.82
E22	18.2 ± 1.3	20.6 ± 2.7	51.2 ± 3.2	0.30 ± 0.08
E23	15.4 ± 1.1	10.5 ± 1.4	46.5 ± 2.9	0.41 ± 0.11
E24	11.7 ± 0.9	2.2 ± 0.3	42.0 ± 2.7	0.10 ± 0.02
E25	32.8 ± 2.4	19.5 ± 2.6	116.9 ± 7.4	2.14 ± 0.55
E26	28.7 ± 2.1	16.8 ± 2.2	127.4 ± 8.0	2.02 ± 0.52
E27	25.4 ± 1.8	8.8 ± 1.2	114.6 ± 7.2	2.56 ± 0.66
E28	22.3 ± 1.6	5.9 ± 0.8	88.5 ± 5.6	0.79 ± 0.20
E29	25.5 ± 1.9	4.4 ± 0.6	95.4 ± 6.0	1.00 ± 0.26
E30	13.7 ± 1.0	1.9 ± 0.3	49.3 ± 3.1	0.06 ± 0.01
E31	20.4 ± 1.5	7.9 ± 1.0	102.0 ± 6.4	0.97 ± 0.25
E32	15.5 ± 1.1	3.0 ± 0.4	47.3 ± 3.0	0.14 ± 0.04
E33	26.6 ± 1.9	3.5 ± 0.5	96.9 ± 6.1	1.30 ± 0.33
E34	25.3 ± 1.8	18.1 ± 2.4	90.1 ± 5.7	1.71 ± 0.44
E35	20.1 ± 1.5	6.2 ± 0.8	94.1 ± 5.9	0.37 ± 0.09
E36	24.0 ± 1.7	3.8 ± 0.5	96.3 ± 6.1	0.20 ± 0.05
K1	22.6 ± 1.6	2.9 ± 0.4	98.6 ± 6.2	0.20 ± 0.05
K2	39.8 ± 2.9	26.4 ± 3.5	142.1 ± 9.0	1.67 ± 0.43

69 Table S5. Paired t-test for spectrophotometric assays.

	Chokeberry		Blueberry		Black goji berry	
	t^*	P	t^*	P	t^*	P
K1 vs E1	2.01	0.14	0.16	0.88	0.13	0.91
K1 vs E2	0.88	0.44	1.59	0.21	1.59	0.21
K1 vs E3	1.14	0.34	1.33	0.27	1.38	0.26
K1 vs E4	1.87	0.16	1.20	0.32	2.48	0.09
K1 vs E5	2.55	0.08	2.48	0.09	2.52	0.09
K1 vs E6	1.94	0.15	1.99	0.14	1.84	0.16
K1 vs E7	2.19	0.12	2.06	0.13	0.45	0.68
K1 vs E8	1.11	0.35	1.34	0.27	1.62	0.20
K1 vs E9	0.17	0.88	0.56	0.61	0.16	0.88
K1 vs E10	1.34	0.27	1.92	0.15	0.62	0.58
K1 vs E11	2.76	0.07	2.52	0.09	3.01	0.06
K1 vs E12	2.17	0.12	2.13	0.12	2.94	0.06
K1 vs E13	2.03	0.14	1.73	0.18	2.98	0.06
K1 vs E14	2.32	0.10	2.41	0.09	3.13	0.05
K1 vs E15	2.17	0.12	2.18	0.12	**3.37**	0.04
K1 vs E16	2.35	0.10	2.37	0.10	3.02	0.06
K1 vs E17	2.32	0.10	3.01	0.06	2.77	0.07
K1 vs E18	2.49	0.09	2.96	0.06	2.66	0.08
K1 vs E19	2.38	0.10	1.55	0.22	1.11	0.35
K1 vs E20	1.38	0.26	0.77	0.50	**3.52**	0.04
K1 vs E21	2.95	0.06	0.71	0.53	**3.20**	0.05
K1 vs E22	1.21	0.31	2.39	0.10	0.61	0.58
K1 vs E23	1.37	0.26	1.70	0.19	0.96	0.41
K1 vs E24	1.89	0.16	1.61	0.21	1.27	0.29
K1 vs E25	2.48	0.09	1.68	0.19	3.17	0.05
K1 vs E26	1.14	0.34	1.11	0.35	2.14	0.12
K1 vs E27	2.27	0.11	0.29	0.79	2.14	0.12
K1 vs E28	1.49	0.23	0.89	0.44	0.60	0.59
K1 vs E29	1.44	0.25	1.22	0.31	0.39	0.72
K1 vs E30	1.41	0.25	1.79	0.17	1.27	0.29
K1 vs E31	1.38	0.26	2.50	0.09	1.11	0.35
K1 vs E32	1.40	0.25	1.21	0.31	1.18	0.32
K1 vs E33	0.11	0.92	1.15	0.33	0.86	0.45
K1 vs E34	2.94	0.06	0.57	0.61	0.56	0.61
K1 vs E35	1.28	0.29	2.09	0.13	0.52	0.64
K1 vs E36	1.43	0.25	0.49	0.66	0.02	0.99

70 *t_{cr}=3.18

Table S5. continued

	Chokeberry		Blueberry		Black goji berry	
	t^*	P	t^*	P	t^*	P
K2 vs E1	1.94	0.15	0.21	0.84	1.91	0.15
K2 vs E2	1.55	0.22	1.63	0.20	1.90	0.15
K2 vs E3	1.85	0.16	1.27	0.29	2.79	0.07
K2 vs E4	2.43	0.09	0.70	0.53	2.31	0.10
K2 vs E5	0.24	0.82	2.28	0.11	0.65	0.56
K2 vs E6	1.85	0.16	1.69	0.19	2.53	0.09
K2 vs E7	1.65	0.20	1.91	0.15	2.02	0.14
K2 vs E8	1.69	0.19	1.73	0.18	1.68	0.19
K2 vs E9	2.30	0.11	0.58	0.60	1.70	0.19
K2 vs E10	1.78	0.17	2.34	0.10	1.42	0.25
K2 vs E11	0.58	0.60	2.37	0.10	0.65	0.56
K2 vs E12	0.03	0.98	2.03	0.14	0.38	0.73
K2 vs E13	1.49	0.23	1.73	0.18	1.49	0.23
K2 vs E14	1.37	0.26	2.23	0.11	0.75	0.51
K2 vs E15	0.12	0.91	2.05	0.13	1.08	0.36
K2 vs E16	1.19	0.32	2.20	0.12	0.74	0.51
K2 vs E17	1.31	0.28	2.75	0.07	0.64	0.57
K2 vs E18	1.12	0.34	2.68	0.08	2.10	0.13
K2 vs E19	1.56	0.22	1.74	0.18	2.83	0.07
K2 vs E20	1.76	0.18	0.35	0.75	1.66	0.20
K2 vs E21	0.85	0.46	0.48	0.66	0.75	0.51
K2 vs E22	1.66	0.20	1.81	0.17	1.44	0.25
K2 vs E23	1.79	0.17	1.77	0.17	1.63	0.20
K2 vs E24	2.18	0.12	1.62	0.20	1.80	0.17
K2 vs E25	1.34	0.27	1.89	0.16	1.77	0.17
K2 vs E26	2.13	0.12	0.61	0.58	2.73	0.07
K2 vs E27	2.24	0.11	0.06	0.96	2.49	0.09
K2 vs E28	1.74	0.18	0.78	0.49	2.10	0.13
K2 vs E29	1.74	0.18	1.23	0.31	2.17	0.12
K2 vs E30	1.80	0.17	1.83	0.16	1.85	0.16
K2 vs E31	1.76	0.18	2.99	0.06	2.44	0.09
K2 vs E32	1.79	0.17	1.14	0.34	1.78	0.17
K2 vs E33	2.54	0.08	1.02	0.38	2.16	0.12
K2 vs E34	2.32	0.10	1.60	0.21	1.63	0.20
K2 vs E35	1.75	0.18	1.75	0.18	2.32	0.10
K2 vs E36	1.74	0.18	0.03	0.98	2.32	0.10
K1 vs K2	2.18	0.12	1.20	0.32	2.47	0.09

*t_{cr}=3.18

Table S6. Extraction repeatability results. Abbreviation: P1-P6- six replicates of NADES blueberry extracts, TPC- total phenolic content, TFC- total flavonoid content, TAC- total anthocyanin content and RSA- radical scavenging activity.

	TPC (g GAE/kg)	TFC (g QUE/kg)	TAC (g cyd-3-glu/kg)	RSA (mol TE/kg)	Anthocyanin Peak area	Phenolic Peak area
P1	13.1	19.9	0.271	65.9	2817	21724
P2	13.9	22.7	0.287	61.5	3368	25499
P3	12.9	19.8	0.273	61.2	2801	24529
P4	12.7	22.4	0.247	60.4	2576	23262
P5	14.0	27.5	0.308	64.6	3503	25061
P6	15.4	20.2	0.466	71.1	4206	29821
Avg.	13.7	22.1	0.309	64.1	3212	24983
SD	1.0	2.9	0.080	4.1	604	2738
RSD	7.3%	13.3%	25.8%	6.3%	18.8%	11.0%

Figure S3. HPTLC profiles of six replicates of NADES blueberry extracts (P1- P6), under VIS and UV light. Zones marked with a yellow square were used to determine repeatability as peak area.

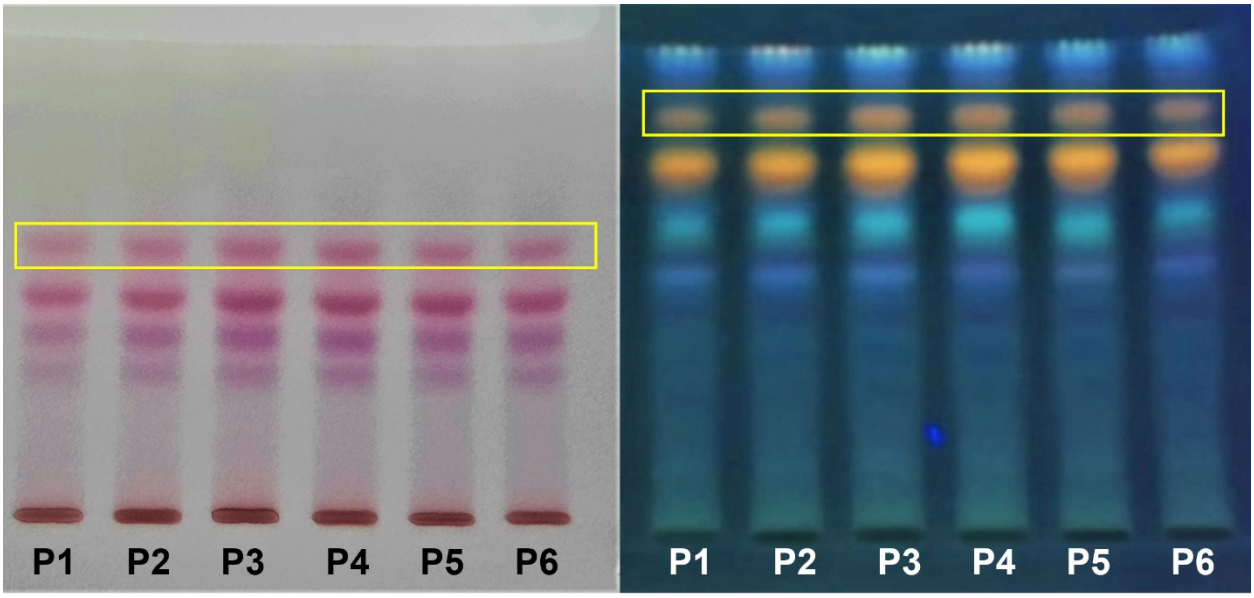

Table S7. Quantified phenolic compounds in selected extracts of each berry.

	Phenolic compounds (mg kg $^{-1}$) *																
	GA	CGA	CA	p-CA	QUE	RUT	QR	HYP	ISR	I3R	I3G	EC	NAR	K3G	MYR	AE	GAL
Chokeberry																	
E5	0.28	870.89	3.17	NF	34.97	187.48	0.22	95.98	2.01	5.85	2.66	33.18	19.55	105.21	NF	NF	NF
E12	1.32	1055.11	8.28	NF	29.44	200.24	0.32	115.19	1.68	6.06	3.17	39.41	84.25	84.24	NF	NF	NF
E14	2.24	1022.82	8.47	NF	23.05	187.94	0.32	121.77	1.24	5.81	2.37	32.50	52.66	76.33	NF	NF	NF
E21	0.18	1007.67	5.39	NF	91.87	180.55	0.22	94.17	1.71	6.32	2.82	31.12	10.74	8.99	NF	NF	NF
E8	0.26	265.18	2.76	NF	6.73	222.97	0.21	129.97	0.34	6.01	2.87	21.82	22.77	44.45	NF	NF	NF
K1	0.91	556.97	41.98	NF	4.10	232.13	0.37	148.99	0.21	7.48	2.84	34.90	53.85	17.18	NF	NF	NF
Blueberry																	
E5	19.97	41.28	4.30	NF	53.39	12.19	0.14	24.22	32.39	3.72	18.97	4.90	NF	3.06	25.50	2.54	NF
E12	21.61	158.80	6.42	NF	61.39	49.76	0.38	41.53	30.95	3.88	14.24	8.69	15.35	9.37	29.62	3.25	NF
E17	15.33	191.03	8.51	NF	7.40	58.22	0.41	49.40	6.75	4.45	8.10	11.57	39.70	11.61	8.55	2.94	NF
E29	10.35	76.54	4.34	NF	31.43	27.64	0.23	32.86	18.75	3.21	5.48	5.44	10.91	5.92	18.42	3.12	NF
E25	16.70	49.66	3.82	NF	46.41	18.80	0.24	30.12	30.35	3.51	6.88	4.13	NF	2.20	8.17	4.42	NF
K1	6.40	64.61	6.06	NF	3.49	37.94	0.47	37.93	1.97	4.72	7.43	5.84	17.13	4.97	5.61	NF	NF
Black goji berry																	
E5	0.97	495.89	10.05	31.42	1.78	162.49	0.09	3.93	2.38	8.81	0.51	205.05	13.64	3.30	2.79	NF	NF
E11	1.79	551.27	7.81	72.40	2.07	178.97	0.20	5.14	3.28	7.71	0.64	245.65	19.02	3.08	2.71	3.81	2.08
E15	1.12	344.87	5.04	27.81	10.67	200.09	0.33	24.63	5.79	9.23	2.16	120.43	49.78	7.85	3.73	3.66	1.93
E21	2.28	418.82	6.83	29.73	1.71	146.61	0.09	4.35	1.92	7.19	0.68	143.99	17.22	2.30	NF	2.72	NF
E34	NF	106.95	3.97	40.90	2.05	186.43	0.14	5.55	2.20	7.46	0.62	89.46	14.93	3.07	3.09	3.33	NF
K1	1.20	685.96	10.77	35.52	1.44	212.27	0.08	4.99	1.33	8.68	0.92	152.18	11.90	2.64	2.60	3.42	NF

*Abbreviations: GA- Gallic acid, CGA- Chlorogenic acid, CA- Caffeic acid, p-CA- p-Coumaric acid, QUE- Quercetin, RUT- Rutin, QR- Quercetin-3-O-rhamnoside Quercitrin, HYP- Hyperoside, ISR- Isorhamnetin, I3R- Isorhametin-3-O-rutinoside, I3G- Isorhametin-3-O-glucoside, EC- Epictechin, NAR- Naringin, K3G- Kaempferol-3-O-glucoside, MYR- Myricetin, AE- Aesculetin, GAL- Galangin, NF- not found.

Table S8. Quantified anthocyanins in selected extracts of each berry.

	Anthocyanins (mg kg $^{-1}$) *										
	D3G	C3S	C3,5-diG	M3,5-diG	C3G	C3R	C3S	C3A	P3O	P3G	M3G
					Chokeberry						
E5	NF	9.26	25.33	0.37	1390.42	9.30	43.30	2375.18	6.29	NF	0.38
E12	NF	9.30	24.52	0.88	1122.84	9.00	42.37	1942.01	5.79	NF	0.65
E14	NF	7.44	20.28	0.52	993.48	7.08	38.72	1734.30	4.72	NF	2.54
E21	NF	2.49	7.00	0.25	56.59	2.41	25.74	68.27	0.76	NF	0.21
E8	NF	5.73	8.32	0.46	440.19	5.61	33.78	905.03	2.76	NF	0.48
K1	NF	4.67	8.73	0.53	157.07	4.50	35.70	234.75	1.93	NF	0.49
					Blueberry						
E5	743.26	0.68	0.71	18.81	18.10	0.64	0.36	53.92	0.20	16.35	917.04
E12	679.81	1.27	2.55	16.70	73.76	1.20	3.54	120.81	0.65	63.19	730.44
E17	500.92	1.61	3.14	4.59	102.61	1.45	5.23	145.37	1.17	93.97	130.60
E29	198.11	0.79	1.64	2.40	34.30	0.73	1.97	44.75	0.50	33.69	46.32
E25	286.36	0.20	2.63	2.73	18.07	0.20	0.55	18.49	0.17	18.25	25.46
K1	409.47	0.60	1.17	5.13	38.50	0.60	2.16	41.98	0.42	36.12	38.19
					Black goji berry						
E5	19.02	13.74	1.59	4.54	2.90	13.46	0.09	1.05	NF	2.89	1.88
E11	25.98	16.00	1.71	5.55	3.04	15.65	0.09	0.96	NF	2.95	2.60
E15	164.69	15.77	2.32	4.49	10.17	15.77	2.32	9.15	NF	9.96	9.02
E21	22.97	10.29	1.13	3.08	2.63	10.19	0.34	1.06	NF	2.68	2.15
E34	36.25	16.03	1.49	4.27	3.55	16.07	0.48	1.59	NF	3.39	4.32
K1	30.23	17.08	1.89	5.63	3.57	16.97	0.27	0.95	NF	3.46	2.96

*Abbreviations: D3G- Delphidin-3-O-glucoside, C3S- Cyanidin-3-O-sophoroside, C3,5-diG- Cyanidin-3,5-di-O-glucoside, M3,5-diG- Malvidin-3,5-di-O-glucoside, C3G- Cyanidin-3-O-glucoside, C3R- Cyanidin-3-O-rutinoside, C3S- Cyanidin-3-O-sambubioside, C3A- Cyanidin-3-O-arabinoside, P3O- Pelargonidin-3-O- glucoside, P3G- Peonidin-3-O-glucoside, M3G- Malvidin-3-O-glucoside, NF- not found.

92 Table S9. Paired t-test for quantified phenolic compounds in selected extracts.

	t^*	P
	Chokeberry	
K1 vs E5	1.51	0.14
K1 vs E12	1.64	0.11
K1 vs E14	1.61	0.12
K1 vs E21	0.11	0.92
K1 vs E8	0.73	0.47
	Blueberry	
K1 vs E5	1.33	0.19
K1 vs E12	1.89	0.07
K1 vs E17	**3.21**	0.003
K1 vs E29	0.73	0.47
K1 vs E25	1.28	0.21
	Black goji berry	
K1 vs E5	1.05	0.30
K1 vs E11	0.21	0.83
K1 vs E15	0.41	0.68
K1 vs E21	1.39	0.18
K1 vs E34	1.14	0.26

*tcr=2.05

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