

Supplementary Materials

Table S1. Values of different bioactive compounds analysed in broths at pH 2.

Component	0 min	3 min	5 min	7 min	10 min	13 min
Antioxidant activity (µM TE/g CBS)	43.9 ± 0.1a	99.8 ± 0.8b	102.2 ± 1.2b	97.9 ± 0.7b	96.8 ± 0.5b	101.1 ± 0.4b
TPC (mg GAE/g CBS)	4.1 ± 0.1a	7.1 ± 0.1b	7.3 ± 0.1b	8.1 ± 0.1b	9.5 ± 0.1c	9.1 ± 0.1c
TFC (mg CE/g CBS)	2.1 ± 0.2a	2.5 ± 0.01a	4.1 ± 0.2b	3.9 ± 0.1b	6.3 ± 0.2c	6.2 ± 0.2c
TTC (mg CE/g CBS)	1.4 ± 0.05a	5.4 ± 0.2b	5.1 ± 0.01b	4.8 ± 0.2b	5.6 ± 0.1b	5.1 ± 0.1b
TSC (mg EE/g CBS)	16.1 ± 0.1a	31.5 ± 0.1b	30.9 ± 0.01b	32.1 ± 0.1b	35.6 ± 0.6b	32.2 ± 0.8b
Catechin (mg/g CBS)	0.03 ± 0.2a	0.07 ± 0.3b	0.07 ± 0.02b	0.07 ± 0.3b	0.06 ± 0.01b	0.04 ± 0.01a
Epicatechin (mg/g CBS)	0.08 ± 0.01a	0.2 ± 0.02b	0.1 ± 0.03b	0.1 ± 0.01b	0.1 ± 0.01b	0.07 ± 0.01a
Theobromine (mg/g CBS)	1.6 ± 0.01a	6.3 ± 0.1b	7.7 ± 0.1c	6.5 ± 0.1b	6.6 ± 0.2b	5.8 ± 0.1b
Caffeine (mg/g CBS)	0.9 ± 0.01a	1.3 ± 0.01a	1.4 ± 0.01a	1.1 ± 0.1a	0.9 ± 0.1a	0.9 ± 0.01a

TE: Trolox equivalent; GAE: Gallic acid equivalent; CE: Catechin equivalent; EE: Escin equivalent. The means followed by different letters within a file means statistic differences ($p < 0.05$) by Tukey's test.

Table S2. Values of different bioactive compounds analysed in broths at pH 3.

Component	0 min	3 min	5 min	7 min	10 min	13 min
Antioxidant activity (µM TE/g CBS)	45.7 ± 0.7a	111.1 ± 1.3b	114.9 ± 2.1b	124.2 ± 1.9b	118.1 ± 2.4b	112.5 ± 1.6b
TPC (mg GAE/g CBS)	4.8 ± 0.1a	7.6 ± 0.7b	7.7 ± 0.1b	8.2 ± 0.4b	9.6 ± 0.1c	9.8 ± 0.3c
TFC (mg CE/g CBS)	1.9 ± 0.1a	4.4 ± 0.4b	3.9 ± 0.1b	5.1 ± 0.1c	6.1 ± 0.2d	5.6 ± 0.01c
TTC (mg CE/g CBS)	1.3 ± 0.01a	3.2 ± 0.1b	2.9 ± 0.2b	4.5 ± 0.2c	4.6 ± 0.1c	4.2 ± 0.01c
TSC (mg EE/g CBS)	13.7 ± 0.7a	31.5 ± 0.6b	30.9 ± 0.9b	29.1 ± 0.6b	29.4 ± 0.9b	29.6 ± 0.6b
Catechin (mg/g CBS)	0.07 ± 0.01a	0.08 ± 0.01a	0.07 ± 0.01a	0.1 ± 0.01a	0.08 ± 0.01a	0.08 ± 0.01a
Epicatechin (mg/g CBS)	0.04 ± 0.01a	0.05 ± 0.01a	0.05 ± 0.01a	0.1 ± 0.01b	0.1 ± 0.01b	0.07 ± 0.02c
Theobromine (mg/g CBS)	1.6 ± 0.1a	5.6 ± 0.2b	7.2 ± 0.1c	8.8 ± 0.4d	8.2 ± 0.2d	7.3 ± 0.01c
Caffeine (mg/g CBS)	0.7 ± 0.01a	1.1 ± 0.01b	1.2 ± 0.01b	1.1 ± 0.01b	1.5 ± 0.01c	1.7 ± 0.1c

TE: Trolox equivalent; GAE: Gallic acid equivalent; CE: Catechin equivalent; EE: Escin equivalent. The means followed by different letters within a file means statistic differences ($p < 0.05$) by Tukey's test.

Table S3. Values of different bioactive compounds analysed in broths at pH 4.

Component	0 min	3 min	5 min	7 min	10 min	13 min
Antioxidant activity (µM TE/g CBS)	46.9 ± 1.2a	119.3 ± 2.3b	137.6 ± 1.4c	159.9 ± 1.6d	187.3 ± 2.2e	152.8 ± 2.8d
TPC (mg GAE/g CBS)	4.1 ± 0.1a	10.1 ± 0.7b	13.7 ± 0.9b	14.3 ± 0.4b	14.5 ± 1.7	13.3 ± 0.6b
TFC (mg CE/g CBS)	2.3 ± 0.01a	5.2 ± 0.1b	7.1 ± 0.4c	8.4 ± 0.1d	8.3 ± 0.6d	7.9 ± 0.1d
TTC (mg CE/g CBS)	1.5 ± 0.1a	4.5 ± 0.1b	4.6 ± 0.1b	4.8 ± 0.1b	6.3 ± 0.2c	5.9 ± 0.2c
TSC (mg EE/g CBS)	17.1 ± 0.8a	37.3 ± 0.9b	45.6 ± 0.9c	42.8 ± 0.9c	52.5 ± 1.8d	50.1 ± 1.4d
Catechin (mg/g CBS)	0.02 ± 0.01a	0.03 ± 0.01a	0.1 ± 0.01b	0.2 ± 0.01b	0.2 ± 0.01b	0.2 ± 0.01b
Epicatechin (mg/g CBS)	0.04 ± 0.0a	0.1 ± 0.01b	0.07 ± 0.01c	0.1 ± 0.01b	0.3 ± 0.01d	0.2 ± 0.1b
Theobromine (mg/g CBS)	0.9 ± 0.1a	5.3 ± 0.1b	6.9 ± 0.4c	7.1 ± 0.6d	7.3 ± 0.9d	9.4 ± 0.2e
Caffeine (mg/g CBS)	1.8 ± 0.01a	2.5 ± 0.1a	3.4 ± 0.1b	3.5 ± 0.1b	3.5 ± 0.1b	3.5 ± 0.1b

TE: Trolox equivalent; GAE: Gallic acid equivalent; CE: Catechin equivalent; EE: Escin equivalent. The means followed by different letters within a file means statistic differences ($p < 0.05$) by Tukey's test.

Table S4. Values of different bioactive compounds analysed in broths at pH 5.

Component	0 min	3 min	5 min	7 min	10 min	13 min
Antioxidant activity ($\mu\text{M TE/g CBS}$)	27.9 \pm 2.1a	102.9 \pm 3.1b	118.2 \pm 1.9c	133.9 \pm 1.7d	136.5 \pm 3.3d	134.3 \pm 1.6d
TPC (mg GAE/g CBS)	4.1 \pm 0.2a	9.1 \pm 0.6b	10.1 \pm 1.2b	10.9 \pm 1.1b	11.3 \pm 1.6b	12.1 \pm 1.1c
TFC (mg CE/g CBS)	1.2 \pm 0.05a	5.8 \pm 0.1b	5.5 \pm 0.2b	6.7 \pm 0.4b	8.9 \pm 0.4c	10.1 \pm 0.9d
TTC (mg CE/g CBS)	1.3 \pm 0.01a	3.6 \pm 0.0b	3.8 \pm 0.3b	4.3 \pm 0.1b	6.1 \pm 0.5c	6.5 \pm 0.4c
TSC (mg EE/g CBS)	14.3 \pm 1.6a	30.9 \pm 1.1b	28.3 \pm 0.9b	30.4 \pm 1.5b	42.1 \pm 1.8b	43.2 \pm 2.1b
Catechin (mg/g CBS)	n.d.	0.03 \pm 0.01a	0.05 \pm 0.01a	0.05 \pm 0.01a	0.06 \pm 0.01b	0.06 \pm 0.0b
Epicatechin (mg/g CBS)	n.d.	0.03 \pm 0.01a	0.03 \pm 0.01a	0.03 \pm 0.01a	0.06 \pm 0.02b	0.05 \pm 0.0b
Theobromine (mg/g CBS)	1.4 \pm 0.05a	6.2 \pm 0.9b	5.7 \pm 0.4c	4.7 \pm 0.2c	4.3 \pm 0.1cd	4.7 \pm 0.4c
Caffeine (mg/g CBS)	1.1 \pm 0.1a	1.3 \pm 0.05a	1.4 \pm 0.01b	1.5 \pm 0.01b	1.7 \pm 0.1c	1.8 \pm 0.1c

TE: Trolox equivalent; GAE: Gallic acid equivalent; CE: Catechin equivalent; EE: Escin equivalent; n.d. Non detected. The means followed by different letters within a file means statistic differences ($p < 0.05$) by Tukey's test.

Table S5. Values of different bioactive compounds analysed in broths at pH 8.

Component	0 min	3 min	5 min	7 min	10 min	13 min
Antioxidant activity ($\mu\text{M TE/g CBS}$)	52.1 \pm 2.5a	120.4 \pm 1.9b	132.9 \pm 2.6c	155.3 \pm 2.4d	153.8 \pm 1.7d	143.8 \pm 2.8e
TPC (mg GAE/g CBS)	5.1 \pm 0.2a	11.5 \pm 0.8b	14.4 \pm 1.7b	14.4 \pm 0.9b	14.6 \pm 0.5b	13.7 \pm 0.9b
TFC (mg CE/g CBS)	2.3 \pm 0.1a	4.6 \pm 0.1b	5.2 \pm 0.5b	6.1 \pm 0.2c	5.7 \pm 0.1c	7.5 \pm 0.3d
TTC (mg CE/g CBS)	1.5 \pm 0.05a	4.5 \pm 0.2b	4.3 \pm 0.1b	4.7 \pm 0.2b	5.1 \pm 0.3b	4.9 \pm 0.1b
TSC (mg EE/g CBS)	14.6 \pm 1.6a	40.9 \pm 1.6b	41.5 \pm 1.1b	41.8 \pm 1.7b	40.8 \pm 1.2b	40.8 \pm 1.6b
Catechin (mg/g CBS)	n.d.	0.04 \pm 0.01a	0.04 \pm 0.01a	0.05 \pm 0.01a	0.06 \pm 0.01a	0.05 \pm 0.02a
Epicatechin (mg/g CBS)	n.d.	0.02 \pm 0.0a	0.03 \pm 0.01a	0.03 \pm 0.01a	0.04 \pm 0.01b	0.05 \pm 0.0b
Theobromine (mg/g CBS)	1.9 \pm 0.1a	3.2 \pm 0.2b	5.5 \pm 0.1c	6.5 \pm 0.6d	5.5 \pm 0.01c	5.4 \pm 1.2c
Caffeine (mg/g CBS)	0.7 \pm 0.03a	1.1 \pm 0.01b	0.9 \pm 0.01b	1.4 \pm 0.01b	1.7 \pm 0.0b	1.6 \pm 0.2b

TE: Trolox equivalent; GAE: Gallic acid equivalent; CE: Catechin equivalent; EE: Escin equivalent. n.d. Non detected. The means followed by different letters within a file means statistic differences ($p < 0.05$) by Tukey's test.

Table S6. Values of different bioactive compounds analysed in broths at pH 10.

Component	0 min	3 min	5 min	7 min	10 min	13 min
Antioxidant activity ($\mu\text{M TE/g CBS}$)	57.2 \pm 1.3a	120.6 \pm 4.1b	148.8 \pm 2.1b	135.4 \pm 1.9b	131.5 \pm 2.2b	126.8 \pm 3.1b
TPC (mg GAE/g CBS)	6.1 \pm 0.2a	12.3 \pm 1.1	11.5 \pm 2.2b	10.3 \pm 1.2c	10.6 \pm 2.1c	10.3 \pm 1.1c
TFC (mg CE/g CBS)	4.1 \pm 0.1a	4.5 \pm 0.6a	5.6 \pm 0.1b	5.3 \pm 0.2b	5.8 \pm 0.1b	5.1 \pm 0.2b
TTC (mg CE/g CBS)	2.6 \pm 0.01a	3.9 \pm 0.2b	4.4 \pm 0.1bc	4.9 \pm 0.1c	3.9 \pm 1.1bc	3.5 \pm 0.6ab
TSC (mg EE/g CBS)	17.6 \pm 0.9a	36.4 \pm 1.8b	39.8 \pm 1.9bc	35.6 \pm 2.5bd	33.5 \pm 1.9e	29.5 \pm 1.8f
Catechin (mg/g CBS)	0.02 \pm 0.0a	0.03 \pm 0.01a	0.07 \pm 0.01b	0.07 \pm 0.02b	0.08 \pm 0.0b	0.07 \pm 0.03b
Epicatechin (mg/g CBS)	0.05 \pm 0.01a	0.06 \pm 0.0ab	0.06 \pm 0.02b	0.07 \pm 0.01b	0.09 \pm 0.01c	0.06 \pm 0.01b
Theobromine (mg/g CBS)	1.9 \pm 0.4a	4.6 \pm 0.6b	4.9 \pm 0.6b	6.2 \pm 0.6c	7.1 \pm 0.5d	6.9 \pm 0.5cd
Caffeine (mg/g CBS)	1.1 \pm 0.2a	1.2 \pm 0.1b	1.3 \pm 0.1b	1.3 \pm 0.2b	1.4 \pm 0.3c	1.9 \pm 0.01d

TE: Trolox equivalent; GAE: Gallic acid equivalent; CE: Catechin equivalent; EE: Escin equivalent. The means followed by different letters within a file means statistic differences ($p < 0.05$) by Tukey's test.