

## Supplementary Data

### **Influence of extraction technology on rapeseed oil functional quality: a study on rapeseed polyphenols**

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#### **LC-MS analysis**

LC-MS/MS analysis was performed on a LC-MS/MS analysis was performed on a AB SCIEX Exion Ultra Performance Liquid Chromatography system equipped with equipped with AB SCIEX Triple TOF 5600+ mass detector. The separation chromatographic column was a reversed-phase Thermo Hypersil GOLD C18 (100 mm × 2.1 mm, 1.9 μm). Elution conditions: phase A was water, and phase B was 0.1% (v:v) acetic acid in acetonitrile. The gradient elution process was: 0-0.5 min, 10% B; 0.5-15

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min, 10-55% B; 15-15.5 min, 55-100% B; 15.5-17 min, 100% B; 17-17.5 min, 100-10% B; and 17.5-20 min, 10% B. Column temperature: 35°C; Flow rate: 0.3 mL/min; Injection volume: 2 µL. The mass spectrometry parameters were set as follows: electrospray ion source (ESI), capillary voltage 4.5 kV, capillary temperature 500°C, spray gas N<sub>2</sub> and collision gas Ar, Ion Source Gas 1 50, Ion Source Gas 2 50, Curtain Gas 35.

### **Method validation**

#### **Standard curves**

The scan mode, molecular weight, structural formula and retention time of the phenolic compounds were shown in Table 1S. The standard curve equation were constructed from a set of concentrations in each standard, involving three replicate measurements. A linear regression with  $r^2 > 0.9826$  was obtained in all relevant ranges (Table 2S).

#### **Limit of detection (LOD) and limit of quantitation (LOQ)**

The signal-to-noise ratio was used to determine the LOD and LOQ. When the signal-to-noise ratio (S/N) is 10, the concentration is determined to be LOQ, and when the signal-to-noise ratio is 3, the concentration is determined to be LOD. The LODs ranged from 0.004 to 0.012 µg/mL for 12 phenolic compounds (Table 2S).

#### **Recovery rate**

Twelve kinds of standards with different contents (0.5 µg/g, 2 µg/g and 10 µg/g ) were added to the rapeseed oil, and then Diol-SPE column was applied to extract polyphenols, finally LC-MS/MS assay was used to determine phenolic compounds contents. The recovery rates were calculated using the calibration concentration (blank

vs sample), and addition contents. The recovery rates ranged from 80.78% to 112.52% (Table 3S).

### **Precisions**

Precisions include intra-day precisions and inter-day precisions, intra-day precision were measured in the same day, while inter-day precision was calculated by measuring each sample for 5 consecutive days. The calculation results of precision were evaluated by relative standard deviation (RSD), and the precision for all the samples were not more than 12% RSD (Table 3S).

**Table 1S** The scan mode, molecular weight, structural formula and retention time of the phenolic compounds

Compound	RT/mi n	Scan mode	m/z	Formula
Ferulic acid	3.24	[M-H] <sup>-</sup>	193.0495	C <sub>10</sub> H <sub>4</sub> O <sub>4</sub>
Caffeic acid	3.29	[M-H] <sup>-</sup>	179.0339	C <sub>9</sub> H <sub>8</sub> O <sub>4</sub>
Sinapine	1.91	[M-H] <sup>-</sup>	311.1727	C <sub>16</sub> H <sub>24</sub> NO <sub>5</sub>
Sinapic acid	5.12	[M-H] <sup>+</sup>	223.0601	C <sub>11</sub> H <sub>12</sub> O <sub>5</sub>
Syringic acid	3.43	[M-H] <sup>-</sup>	197.0445	C <sub>9</sub> H <sub>10</sub> O <sub>5</sub>
Cinnamic acid	8.57	[M-H] <sup>-</sup>	147.0441	C <sub>9</sub> H <sub>8</sub> O <sub>2</sub>
Vanillic acid	3.29	[M-H] <sup>-</sup>	167.0339	C <sub>8</sub> H <sub>8</sub> O <sub>4</sub>
p-Coumaric acid	4.59	[M-H] <sup>-</sup>	163.0390	C <sub>9</sub> H <sub>8</sub> O <sub>3</sub>
Gallic acid	1.26	[M-H] <sup>-</sup>	169.0132	C <sub>7</sub> H <sub>6</sub> O <sub>5</sub>
Chlorogenic acid	4.59	[M-H] <sup>-</sup>	353.0867	C <sub>16</sub> H <sub>18</sub> O <sub>9</sub>
Salicylic acid	6.27	[M-H] <sup>-</sup>	137.0233	C <sub>7</sub> H <sub>6</sub> O <sub>3</sub>
p-Hydroxybenzoic acid	2.78	[M-H] <sup>-</sup>	137.0233	C <sub>7</sub> H <sub>6</sub> O <sub>3</sub>

**Table 2S** Linear equation, R<sup>2</sup>, linear ranges, LODs, and LOQs of the phenolic compounds

Compound	Linear range	R <sup>2</sup>	Range (µg/mL)	LOD (µg/mL)	LOQ (µg/mL)
Ferulic acid	$y = 13.36x + 25.24$	0.9904	0.04-80	0.004	0.013
Caffeic acid	$y = 37.02x - 31.15$	0.9976	0.04-80	0.012	0.040
Sinapine	$y = 1.38x - 10.20$	0.9826	0.04-80	0.006	0.019
Sinapic acid	$y = 30.56x + 12.35$	0.9835	0.04-80	0.006	0.019
Syringic acid	$y = 17.70x + 13.84$	0.9866	0.04-80	0.004	0.013
Cinnamic acid	$y = 24.27x - 21.26$	0.9913	0.04-80	0.004	0.013
Vanillic acid	$y = 10.56x + 42.95$	0.9895	0.04-80	0.006	0.019
p-Coumaric acid	$y = 38.13x - 19.03$	0.9833	0.04-80	0.008	0.027
Gallic acid	$y = 10.20x - 26.67$	0.9917	0.04-80	0.012	0.040
Chlorogenic acid	$y = 9.07x + 7.07$	0.9868	0.04-80	0.004	0.013
Salicylic acid	$y = 19.54x - 37.81$	0.9838	0.04-80	0.006	0.019
p-Hydroxybenzoic acid	$y = 7.13x - 4.32$	0.9933	0.04-80	0.004	0.013

**Table 3S** Recovery rate and precision of the phenolic compounds

Compound	Adding standard matter amount ( $\mu\text{g/g}$ )	Recovery rate (%)	RSD %	
			Intra-day precision	Inter-day precision
Ferulic acid	0.5	85.87	1.26	3.14
	2	90.54	2.44	4.96
	10	93.94	2.35	9.42
Caffeic acid	0.5	75.48	2.93	5.54
	2	83.59	2.73	4.27
	10	85.84	1.91	6.62
Sinapine	0.5	108.76	1.86	7.12
	2	112.52	4.71	7.34
	10	112.36	2.42	8.29
Sinapic acid	0.5	92.37	1.61	5.79
	2	95.14	5.08	5.70
	10	96.15	6.01	9.69
Syringic acid	0.5	75.83	7.16	10.05
	2	72.98	1.88	7.31
	10	83.48	4.28	6.88
Cinnamic acid	0.5	84.96	2.68	10.28
	2	92.61	3.44	7.69
	10	96.10	4.21	5.28
Vanillic acid	0.5	80.39	1.81	3.91
	2	87.31	8.91	11.34
	10	100.51	5.74	6.29
p-Coumaric acid	0.5	80.78	2.61	4.72
	2	89.42	7.70	11.23
	10	96.62	4.51	9.84
Gallic acid	0.5	83.01	6.94	8.54
	2	95.36	5.62	7.18
	10	97.28	1.60	6.67
Chlorogenic acid	0.5	79.23	4.61	11.32
	2	83.58	3.81	7.21
	10	88.64	1.73	4.18
Salicylic acid	0.5	89.20	3.76	5.87
	2	89.70	4.23	5.95
	10	96.12	1.81	3.78
p-Hydroxybenzoic acid	0.5	88.25	4.50	5.18
	2	89.76	4.61	5.04
	10	97.83	2.40	5.76