

Electronic Supplementary Information

Preparation of spiked samples for optimization of PLE conditions

Spiked samples were prepared by adding appropriate amounts of eight standards solutions to dry blank dregs, and the final contents of the standards in the spiked samples were: piceid, 5 mg/g; 2,3,5,4'-tetrahydroxystilbene-2-O- β -D-glucoside, 15 mg/g; rhaponticoside, 10 mg/g; emodin-8-glucoside, 3 mg/g; physcion-8-glucoside, 1 mg/g; emodin, 0.5 mg/g; chrysophanol, 2 mg/g; physcion, 2 mg/g. After drying, the spiked samples (0.2 g) was mixed with diatomaceous earth (0.5 g) and placed into 11-mL stainless steel extraction cell, respectively. Extraction of the spiked samples according to the orthogonal test $L_9(3^4)$ (Table S1). The extract was transferred into a 25-mL volumetric flask, which was filled up to the calibration mark with methanol. The test solution was determined with the present HPLC method, and the total determined amount of the spiked standards was calculated using the calibration curves in Table 3. The recovery=100*(total amount of the determined standards/ total amount of the spiked standards). From the results in Table S2, the condition of A3B1C2D2 (namely extraction temperature, 150 °C; pressure, 1200 psi; time, 15 min and flush volume, 45%) was chosen as the optimized PLE conditions.

Table S1 Factors and levels

Levels	A: Temperature (°C)	B: Pressure (psi)	C: Time (min)	D: Flush volume (%)
1	100	1200	10	30
2	120	1500	15	45
3	150	1800	20	60

Table S2 Results of $L_9(3^4)$ orthogonal test

Test No.	Four factors				Recovery of the spiked compounds (%)
	A	B	C	D	
1	1	1	1	1	65
2	1	2	2	2	67
3	1	3	3	3	68
4	2	1	2	3	70
5	2	2	3	1	59
6	2	3	1	2	67
7	3	1	3	2	89
8	3	2	1	3	75
9	3	3	2	1	80
<i>K1</i>	200	224	207	204	
<i>K2</i>	196	201	217	223	
<i>K3</i>	244	215	216	213	
Extreme deviation	48	23	10	19	