

1 **Optimization of an Ultrasound-assisted extraction for simultaneous**
2 **determination of antioxidants in sesame with response surface**
3 **methodology**

4 Dandan Wang^{1,2,†}, Liangxiao Zhang^{1,3,4,†}, Yueqing Xu^{1,2}, Xin Qi^{1,4}, Xuefang Wang^{1,4}, Xiupin
5 Wang^{1,4}, Qi Zhang^{1,5}, Peiwu Li^{1,3,4,5,*}

6 ¹ Oil Crops Research Institute, Chinese Academy of Agricultural Sciences, Wuhan 430062,
7 China

8 ² Key Laboratory of Biology and Genetic Improvement of Oil Crops, Ministry of Agriculture
9 and Rural Affairs, Wuhan 430062, China

10 ³ Laboratory of Quality and Safety Risk Assessment for Oilseed Products (Wuhan), Ministry
11 of Agriculture and Rural Affairs, Wuhan 430062, China

12 ⁴ Quality Inspection and Test Center for Oilseed Products, Ministry of Agriculture and Rural
13 Affairs, Wuhan 430062, China

14 ⁵ Key Laboratory of Detection for Mycotoxins, Ministry of Agriculture and Rural Affairs,
15 Wuhan 430062, China

16 [†] These authors contributed equally to this study

17

18 *Corresponding authors: prof. Peiwu Li at Oil Crops Research Institute, Chinese Academy of
19 Agricultural Sciences, Wuhan 430062, China

20 Tel.: +86 27 86812943; Fax: +86 27 86812862

21 E-mail addresses: Peiwuli@oilcrops.cn (P. Li)

22

23 **Supplementary Materials**

24 Figure S1 Results of preliminary screening on six general parameters

25 Figure S2 LC-MS/MS chromatograms obtained for the standard solution of 10 targets

26 Figure S3 Box plot of differential compounds

27 Table S1 Factors and coded value of Plackett-Burman design

28 Table S2 Design matrix and results of Plackett-Burman test

29 Table S3 Factors and coded value of Box-Behnken design

30 Table S4 Design matrix and results of Box-Behnken test

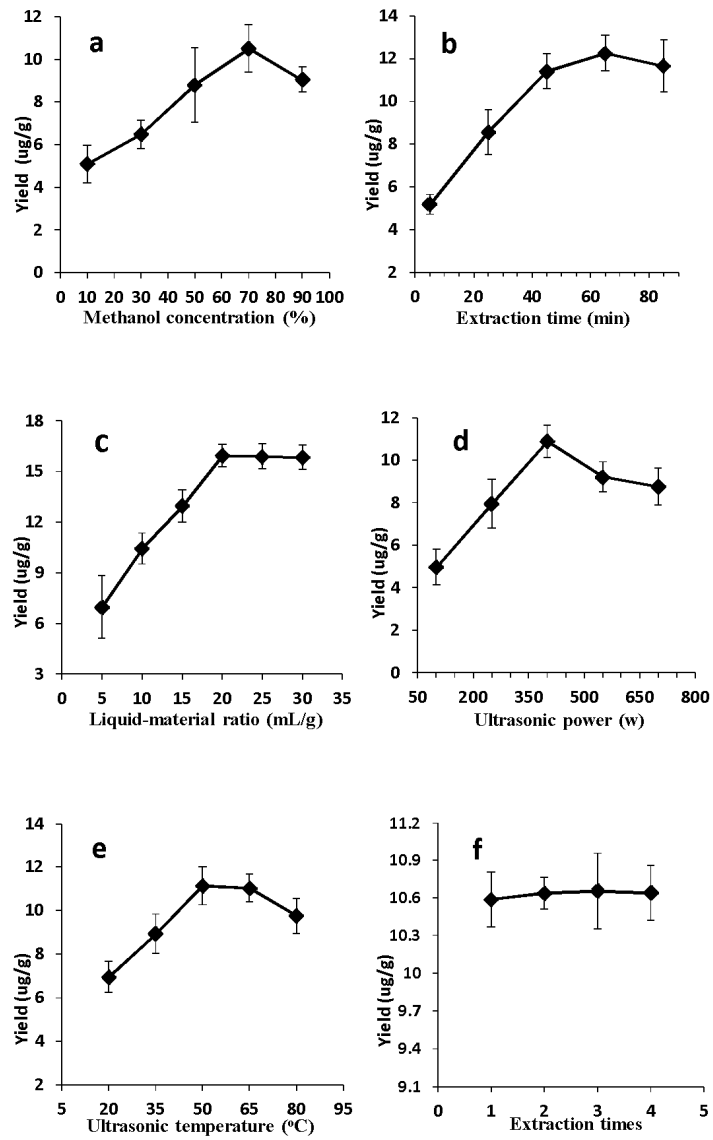
31 Table S5 Analysis results of Plackett-Burman design

32 Table S6 Analysis results of Box-Behnken design

33

34

35 Figure S1 Results of preliminary screening on six general parameters



36

37

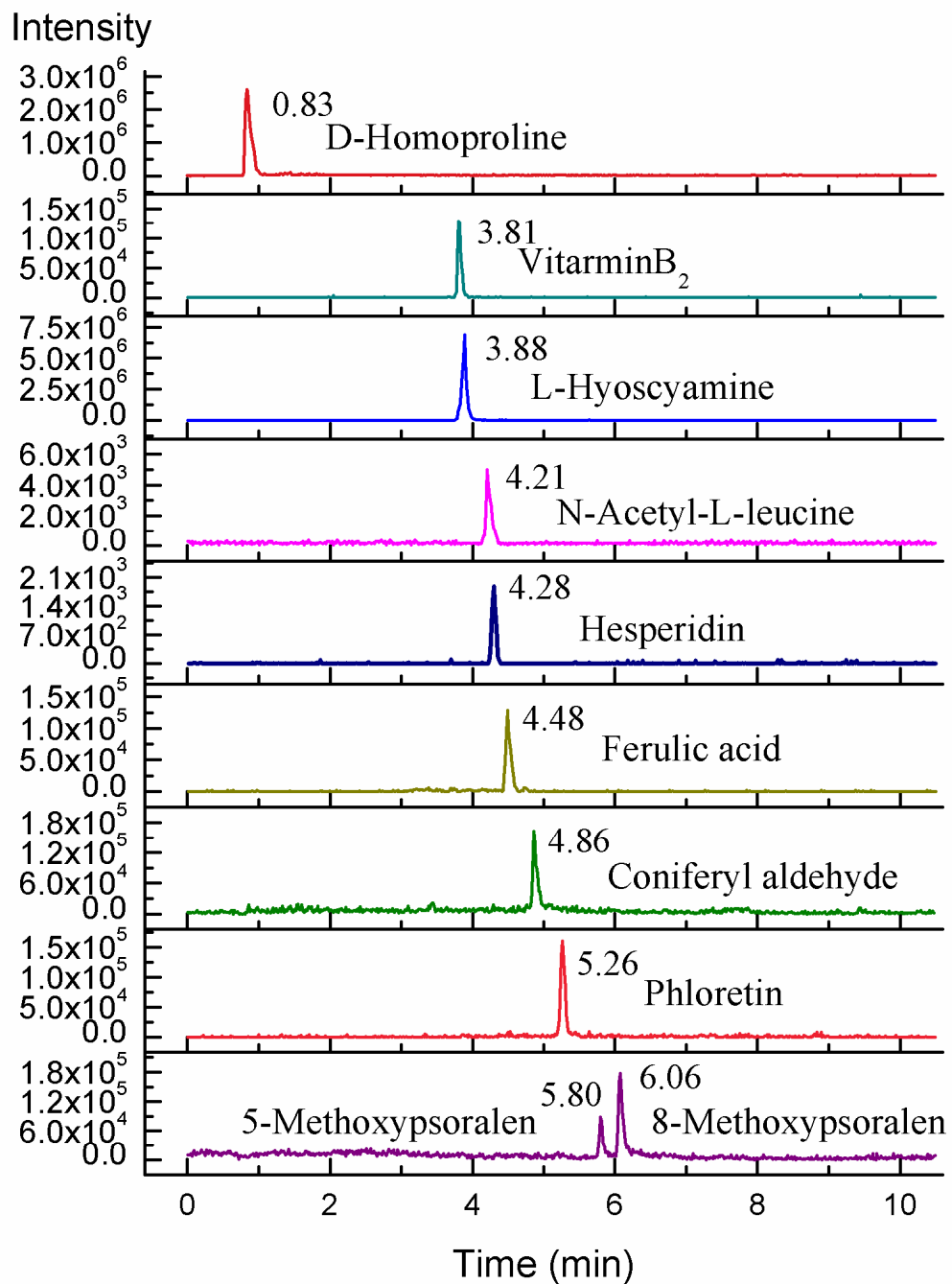
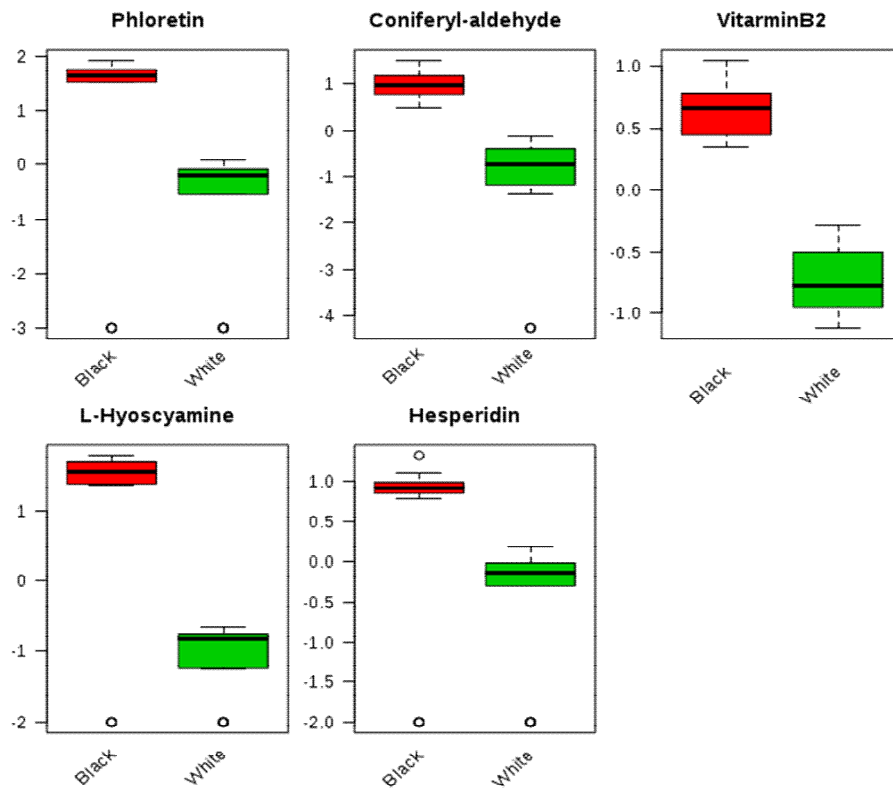


Figure S3 Box plot of differential compounds



44 **Table S1 Factors and coded value of Plackett-Burman design**

Term	Variable	Coded level	
		-1	1
X ₁	liquid-solid ratio (mL/g)	15/1	25/1
X ₂	ultrasonic power (w)	300	500
X ₃	ultrasonic time (min)	35	55
X ₄	ultrasonic temperature (°C)	40	60
X ₅	methanol volume fraction (%)	70	90
X ₆	extraction times	1	3

45

46

Table S2 Design matrix and results of Plackett-Burman test

Run	Liquid-solid ratio (X ₁)	Ultrasonic power (X ₂)	Ultrasonic time (X ₃)	Ultrasonic temperature (X ₄)	Methanol volume fraction (X ₅)	Extraction times (X ₆)	Total actual yield (ug/g)
1	-1	-1	-1	-1	1	1	9.265
2	1	1	-1	1	1	1	9.530
3	1	-1	-1	1	-1	-1	17.518
4	-1	-1	1	1	1	1	10.183
5	1	1	1	-1	1	-1	9.346
6	-1	1	-1	1	1	-1	9.095
7	-1	1	-1	-1	-1	-1	11.596
8	-1	1	1	-1	-1	1	11.959
9	1	-1	-1	-1	-1	1	17.888
10	1	1	1	1	-1	1	16.433
11	1	-1	1	-1	1	-1	11.165
12	-1	-1	1	1	-1	-1	13.915

47

48

49

50

Table S3 Factors and coded value of Box-Behnken design

Term	Variable	Coded level		
		-1	0	1
X ₁	liquid-solid ratio (mL/g)	15/1	20/1	25/1
X ₂	ultrasonic power (w)	300	400	500
X ₅	methanol volume fraction (%)	70	90	90

51

52

53

54

Table S4 Design matrix and results of Box-Behnken test

Run	Liquid-solid ratio (X ₁)	Ultrasonic power (X ₂)	Methanol volume fraction (X ₅)	Total actual yield (ug/g)
1	-1	-1	0	12.322
2	1	-1	0	15.381
3	-1	1	0	12.896
4	1	1	0	17.828
5	-1	0	-1	17.303
6	1	0	-1	19.937
7	-1	0	1	15.013
8	1	0	1	17.326
9	0	-1	-1	16.694
10	0	1	-1	17.912
11	0	-1	1	14.298
12	0	1	1	15.977
13	0	0	0	21.135
14	0	0	0	21.537
15	0	0	0	21.308

Table S5 Analysis results of Plackett-Burman design

Regression data					
Variable	Degree of freedom	Parameter Estimate	Standard Error	t Value	Pr > t
Intercept	1	12.32442	0.30139	40.89	0.0005**
X ₁	1	1.32225	0.30139	4.39	0.0071**
X ₂	1	-0.99792	0.30139	-3.31	0.0212*
X ₃	1	-0.15758	0.30139	-0.52	0.6234
X ₄	1	0.45458	0.30139	1.51	0.1918
X ₅	1	-2.56042	0.30139	-8.50	0.0004**
X ₆	1	0.21858	0.30139	0.73	0.5008
Analysis of Variance					
Source	Degree of freedom	Sum of squares	Mean square	F Value	Pr > F
Model	6	114.95008	19.15835	17.58	0.0032**
Error	5	5.45002	1.09000		
Corrected Total	11	120.40010			
Root MSE	1.04403	R-Square	0.9547		
Dependent Mean	12.32442	Adj R-Sq	0.9004		
Coeff Var	8.47126				

*,** represent significant differences (P<0.05) and extremely significant differences (P<0.01),

respectively.

Table S6 Analysis results of Plackett-Burman design

Source	Degree of freedom	Sum of squares	Mean Square	F Value	Pr > F
Linear	3	35.95549	0.2994	46.74	0.0004**
Quadratic	3	81.893687	0.6820	106.46	<.0001**
Crossproduct	3	0.955923	0.0080	1.24	0.3870
Total Model	9	118.805159	0.9893	51.48	0.0002**
Lack of Fit	3	1.200776	0.400259	9.84	0.0936
Pure Error	2	0.081325	0.040662		
Total Error	5	1.282101	0.256420		
Root MSE	0.506380	R-Square	0.9893	Coefficient of Variation	2.9571
Variable	Degree of freedom	Parameter Estimate	Standard Error	t Value	Pr > t
Intercept	1	-151.965333	22.074508	-6.88	0.0010**
X ₁	1	4.513583	0.619841	7.23	0.0008**
X ₂	1	0.295224	0.030992	9.53	0.0002**
X ₅	1	172.533333	44.566674	3.87	0.0117*
X ₁ *X ₁	1	-0.110908	0.010541	-10.52	<.0001**
X ₂ *X ₁	1	0.000936	0.000506	1.85	0.1236
X ₂ *X ₂	1	-0.000395	0.000026353	-14.98	<.0001**
X ₅ *X ₁	1	-0.160500	0.506380	-0.32	0.7641
X ₅ *X ₂	1	0.011525	0.025319	0.46	0.6681
X ₅ *X ₅	1	-115.920833	26.352827	-4.40	0.0070**

*,** represent significant differences (P<0.05), extremely significant differences (P<0.01),

respectively; F listed value (95%), F_{9,5} = 3.63.