

Supplementary Material for

**Green Extraction Techniques for Obtaining Bioactive
Compounds from Mandarin Peel (*Citrus unshiu* var. *Kuno*):
Phytochemical Analysis and Process Optimization**

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Figure captions:

Figure S1. HPLC chromatograms (selected sample 8 sample 17).

Figure S2. Comparison of UV spectra of standards and unknown peaks; A) hesperidin, B) narirutin, C) rutin, D) 5-HMF, and E) chlorogenic acid.

Figure S3. 3D diagrams of the effects of process parameters on the formation of narirutin in SWE extracts of mandarin peel (*Citrus unshiu* var. *Kuno*).

Figure S4. 3D diagrams of the effects of process parameters on the formation of rutin in SWE extracts of mandarin peel (*Citrus unshiu* var. *Kuno*).

Figure S5. 3D diagram of the effects of process parameters on the formation of chlorogenic acid in SWE extracts of mandarin peel (*Citrus unshiu* var. *Kuno*).

Figure S1

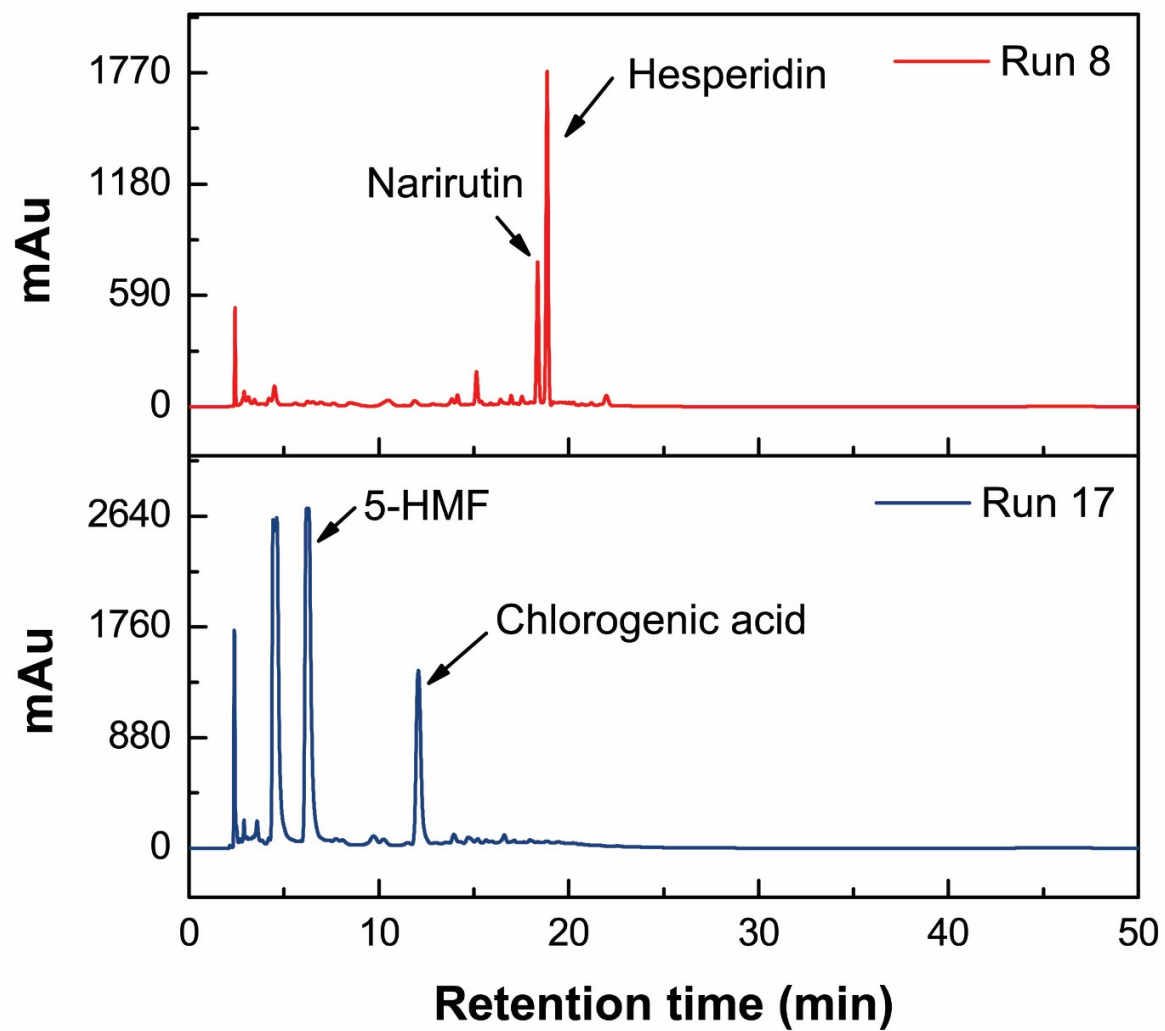


Figure S2

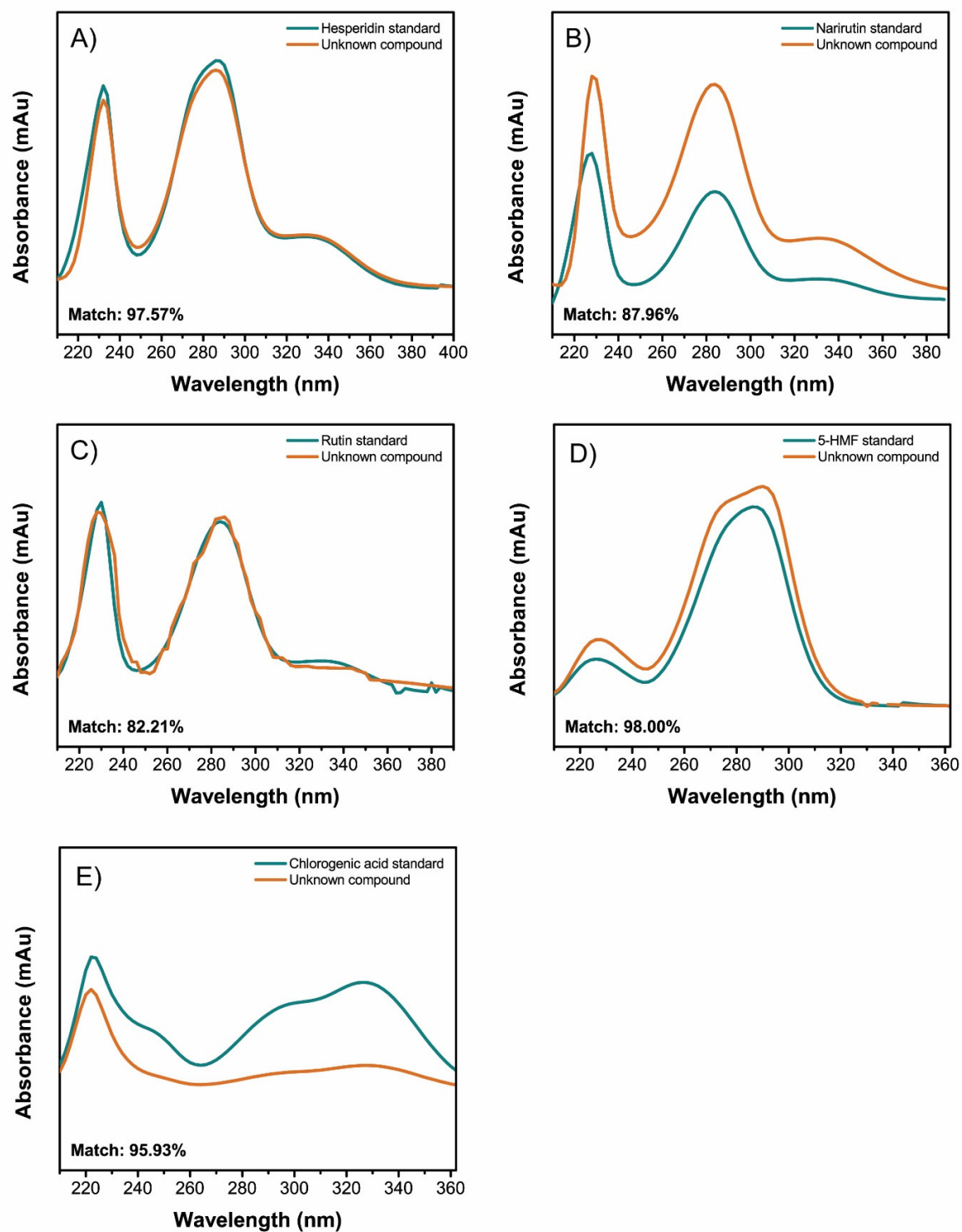


Figure S3

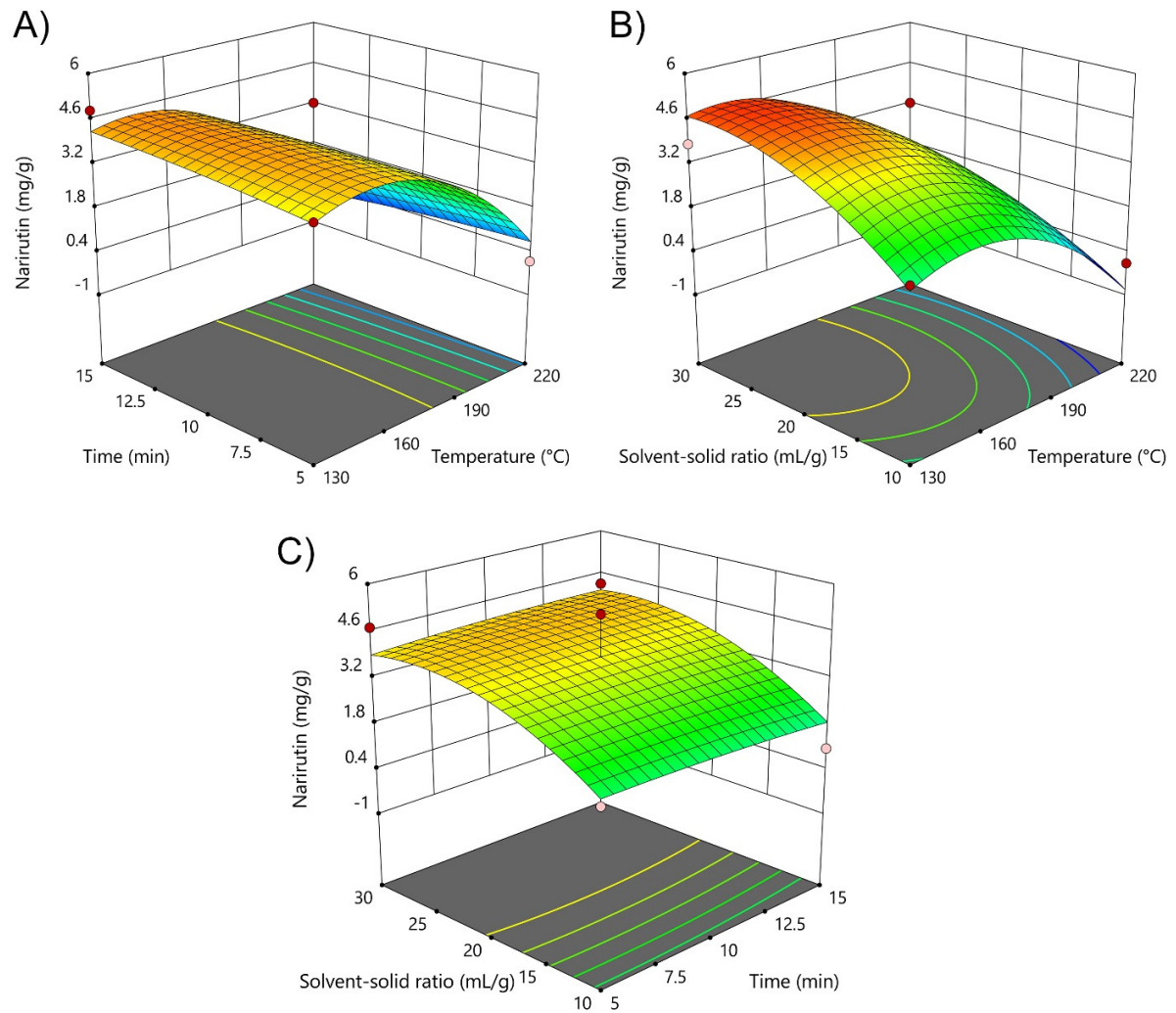


Figure S4

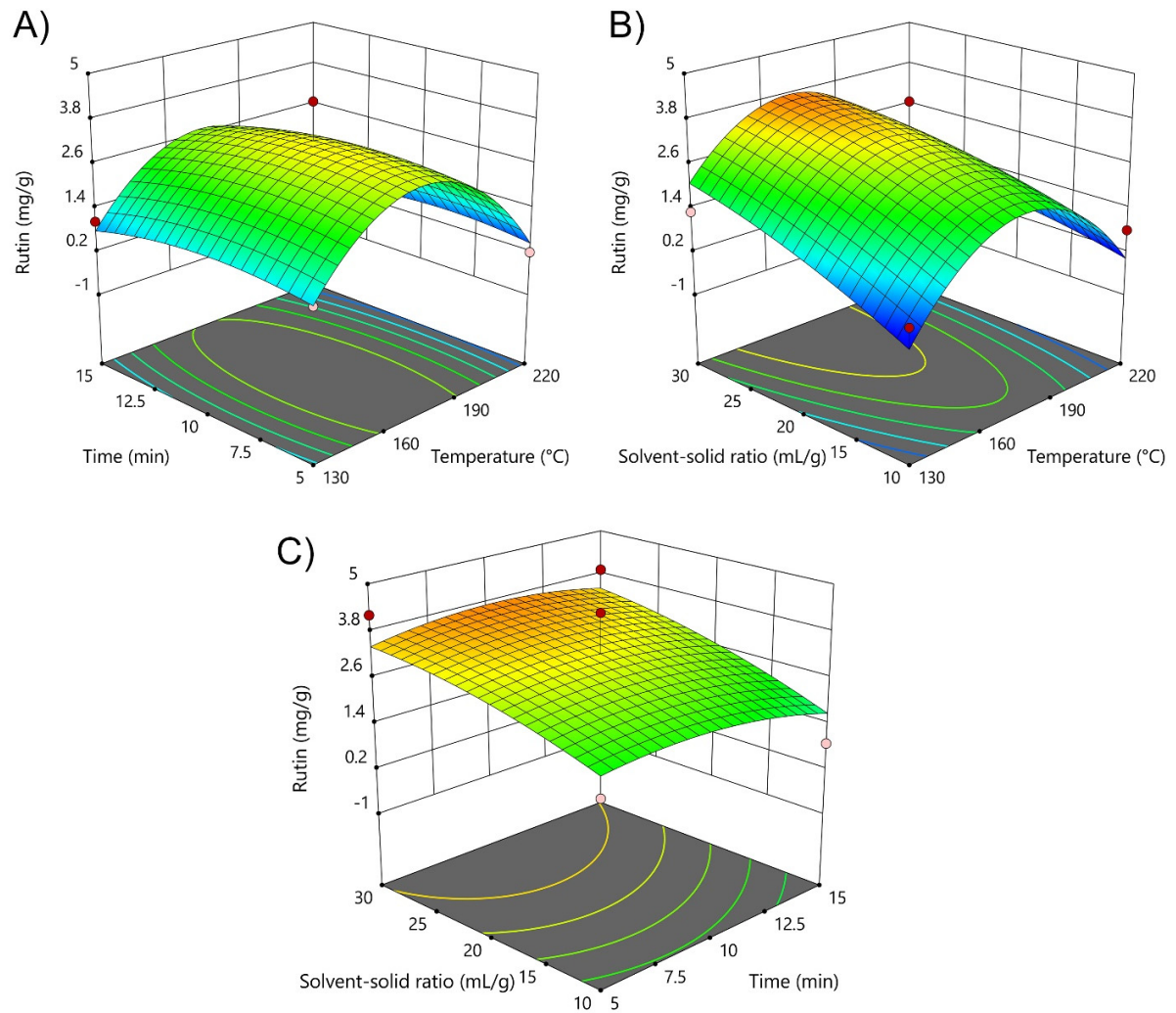


Figure S5

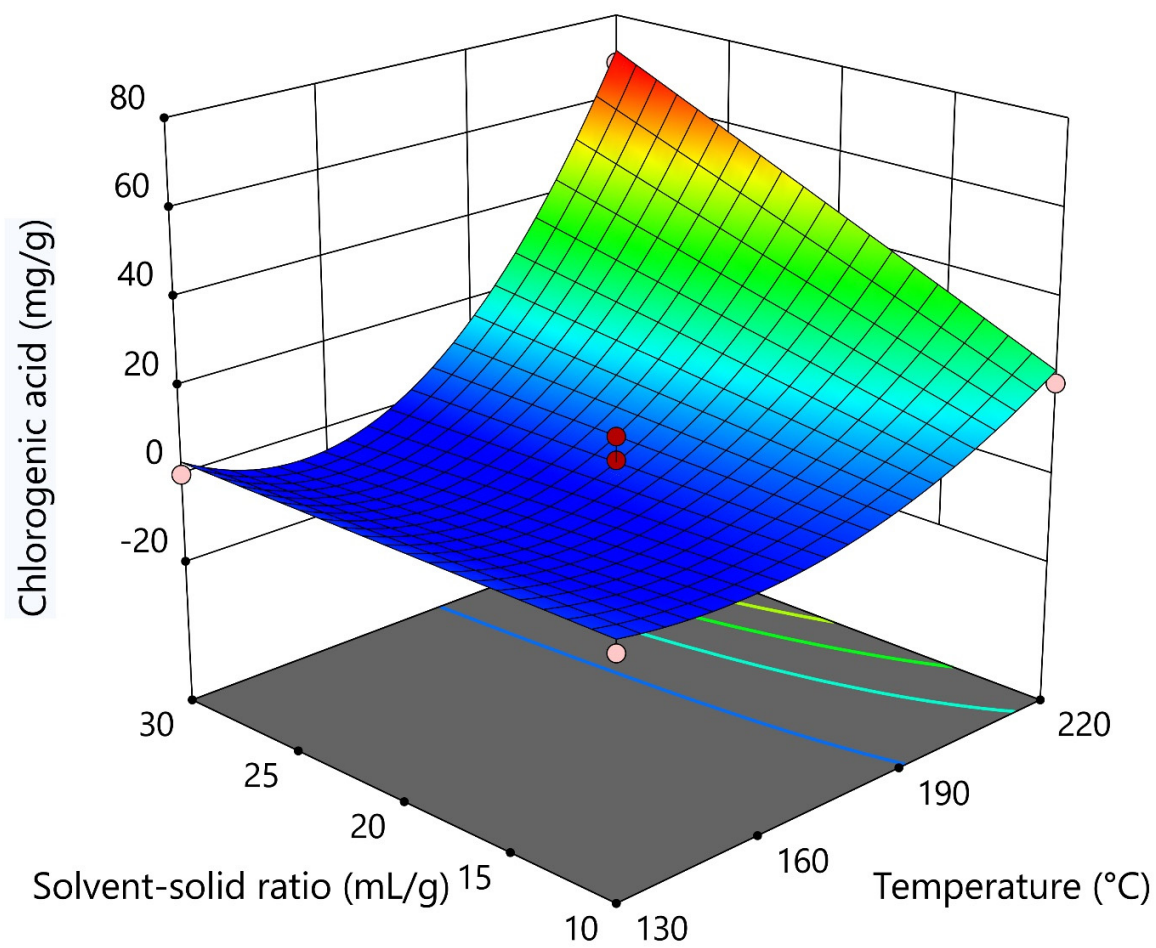


Table S1. Analysis of variance (ANOVA) of second-order polynomial models for 5-HMF content in the mandarin peels (*Citrus unshiu* var. *Kuno*) obtained by SWE technique.

Source	Sum of Squares	Degree of Freedom (df)	Mean Square	F Value	p-Value ^a
Model	241.09	9	26.79	23.29	0.0002
X_1 - temperature	163.24	1	163.24	141.91	<0.0001
X_2 - time	0.2552	1	0.2552	0.22	0.6520
X_3 - solvent-solid ratio	41.72	1	41.72	36.27	0.0005
X_1X_2	3.35	1	3.35	2.91	0.1316
X_1X_3	19.95	1	19.95	17.35	0.0042
X_2X_3	0.25	1	0.25	0.22	0.6553
X_1^2	10.93	1	10.93	9.50	0.0177
X_2^2	0.20	1	0.20	0.17	0.6886
X_3^2	1.45	1	1.45	1.26	0.2984
Residual	8.05	7	1.15		
Lack of fit	5.88	3	1.96	3.60	0.1238
Pure error	2.18	4	0.5439		
Total	249.15	16			
R^2	0.9677				

^a $p < 0.01$ highly significant; $0.01 \leq p < 0.05$ significant; $p \geq 0.05$ not significant.

Table S2. Analysis of variance (ANOVA) of second-order polynomial models for hesperidin content in the mandarin peels (*Citrus unshiu* var. *Kuno*) obtained by SWE technique.

Source	Sum of Squares	Degree of Freedom (df)	Mean Square	F Value	p-Value ^a
Model	371.66	9	41.30	10.61	0.0026
X_1 - temperature	109.44	1	109.44	28.11	0.0011
X_2 - time	6.10	1	6.10	1.57	0.2510
X_3 - solvent-solid ratio	54.30	1	54.30	13.95	0.0073
X_1X_2	3.87	1	3.87	0.9942	0.3519
X_1X_3	10.05	1	10.05	2.58	0.1522
X_2X_3	2.98	1	2.98	0.7649	0.4108
X_1^2	182.13	1	182.13	46.78	0.0002
X_2^2	2.59	1	2.59	0.6663	0.4412
X_3^2	0.55	1	0.5538	0.1422	0.7172
Residual	27.26	7	3.89		
Lack of fit	22.23	3	7.41	5.90	0.0597
Pure error	5.03	4	1.26		
Total	398.91	16			
R^2	0.9317				

^a $p < 0.01$ highly significant; $0.01 \leq p < 0.05$ significant; $p \geq 0.05$ not significant.

Table S3. Analysis of variance (ANOVA) of second-order polynomial models for narirutin content in the mandarin peels (*Citrus unshiu* var. *Kuno*) obtained by SWE technique.

Source	Sum of Squares	Degree of Freedom (df)	Mean Square	F Value	p-Value
Model	47.72	9	5.30	6.35	0.0118
X_1 - temperature	24.99	1	24.99	29.90	0.0009
X_2 - time	0.0058	1	0.0058	0.0069	0.9362
X_3 - solvent-solid ratio	7.90	1	7.90	9.45	0.0180
X_1X_2	0.3102	1	0.3102	0.3712	0.5616
X_1X_3	0.7676	1	0.7676	0.9187	0.3696
X_2X_3	0.0436	1	0.0436	0.0522	0.8258
X_1^2	10.36	1	10.36	12.40	0.0097
X_2^2	0.0239	1	0.0239	0.0286	0.8705
X_3^2	2.58	1	2.58	3.09	0.1221
Residual	5.85	7	0.8356		
Lack of fit	3.79	3	1.26	2.46	0.2026
Pure error	2.06	4	0.5142		
Total	53.57	16			
R^2	0.8908				

^a $p < 0.01$ highly significant; $0.01 \leq p < 0.05$ significant; $p \geq 0.05$ not significant.

Table S4. Analysis of variance (ANOVA) of second-order polynomial models for rutin content in the mandarin peels (*Citrus unshiu* var. *Kuno*) obtained by SWE technique.

Source	Sum of Squares	Degree of Freedom (df)	Mean Square	F Value	p-Value ^a
Model	29.11	9	3.23	4.36	0.0325
X_1 - temperature	0.7642	1	0.7642	1.03	0.3437
X_2 - time	0.1556	1	0.1556	0.21	0.6607
X_3 - solvent-solid ratio	4.20	1	4.20	5.67	0.0489
X_1X_2	0.00	1	0.00	0.00	0.9964
X_1X_3	0.4424	1	0.4424	0.5969	0.4650
X_2X_3	0.0605	1	0.0605	0.0817	0.7833
X_1^2	22.01	1	22.01	29.70	0.0010
X_2^2	0.4767	1	0.4767	0.6432	0.4489
X_3^2	0.2183	1	0.2183	0.2946	0.6042
Residual	5.19	7	0.7411		
Lack of fit	3.79	3	1.26	3.61	0.1233
Pure error	1.40	4	0.3496		
Total	34.29	16			
R^2	0.8487				

^a $p < 0.01$ highly significant; $0.01 \leq p < 0.05$ significant; $p \geq 0.05$ not significant.

Table S5. Analysis of variance (ANOVA) of reduced third-order polynomial models for chlorogenic acid content in the mandarin peels (*Citrus unshiu* var. *Kuno*) obtained by SWE technique.

Source	Sum of Squares	Degree of Freedom (df)	Mean Square	F Value	p-Value ^a
Model	8469.29	8	1058.66	63.19	< 0.0001
X_I -temperature	2001.42	1	2001.42	119.47	< 0.0001
X_1X_3	555.10	1	555.10	33.13	0.0004
X_1^2	2111.05	1	2111.05	126.01	< 0.0001
$X_1^2X_3$	564.58	1	564.58	33.70	0.0004
$X_1X_2^2$	69.88	1	69.88	4.17	0.0754
$X_2^2X_3$	9.87	1	9.87	0.5893	0.4648
$X_2X_3^2$	25.46	1	25.46	1.52	0.2527
X_2^3	5.64	1	5.64	0.3364	0.5779
Residual	134.02	8	16.75		
Lack of fit	87.19	4	21.80	1.86	0.2810
Pure error	46.83	4	11.71		
Total	8603.31	16			
R^2	0.9844				

^a p < 0.01 highly significant; 0.01 ≤ p < 0.05 significant; p ≥ 0.05 not significant.