

Supplementary Information

Occurrence of Pesticides in Pericarpium Citri Reticulatae and Related Products Using Syringe Filter-Based Cleanup

*Fuxin Liu,^{a,#} Xiaowen Dou,^{b,#} Jiaoyang Luo,^b Dandan Kong,^b Zhuowen Fan^{*a} and Meihua Yang^{*b}*

^aCollege of Pharmacy, Heilongjiang University of Chinese Medicine, 150040 Harbin, Heilongjiang, China

^bKey Laboratory of Bioactive Substances and Resources Utilization of Chinese Herbal Medicine, Ministry of Education, Institute of Medicinal Plant Development, Chinese Academy of Medical Sciences, Peking Union Medical College, 100193 Beijing, China

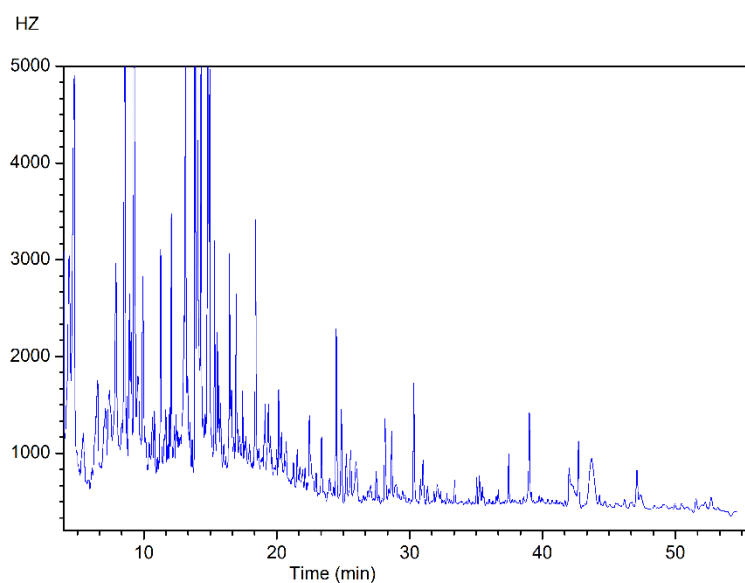


Figure S1. GC-ECD chromatogram of negative dried tangerine peel extract without purification.

*e-mail: 1282776014@qq.com; yangmeihua15@hotmail.com

#These authors contributed equally to this work.

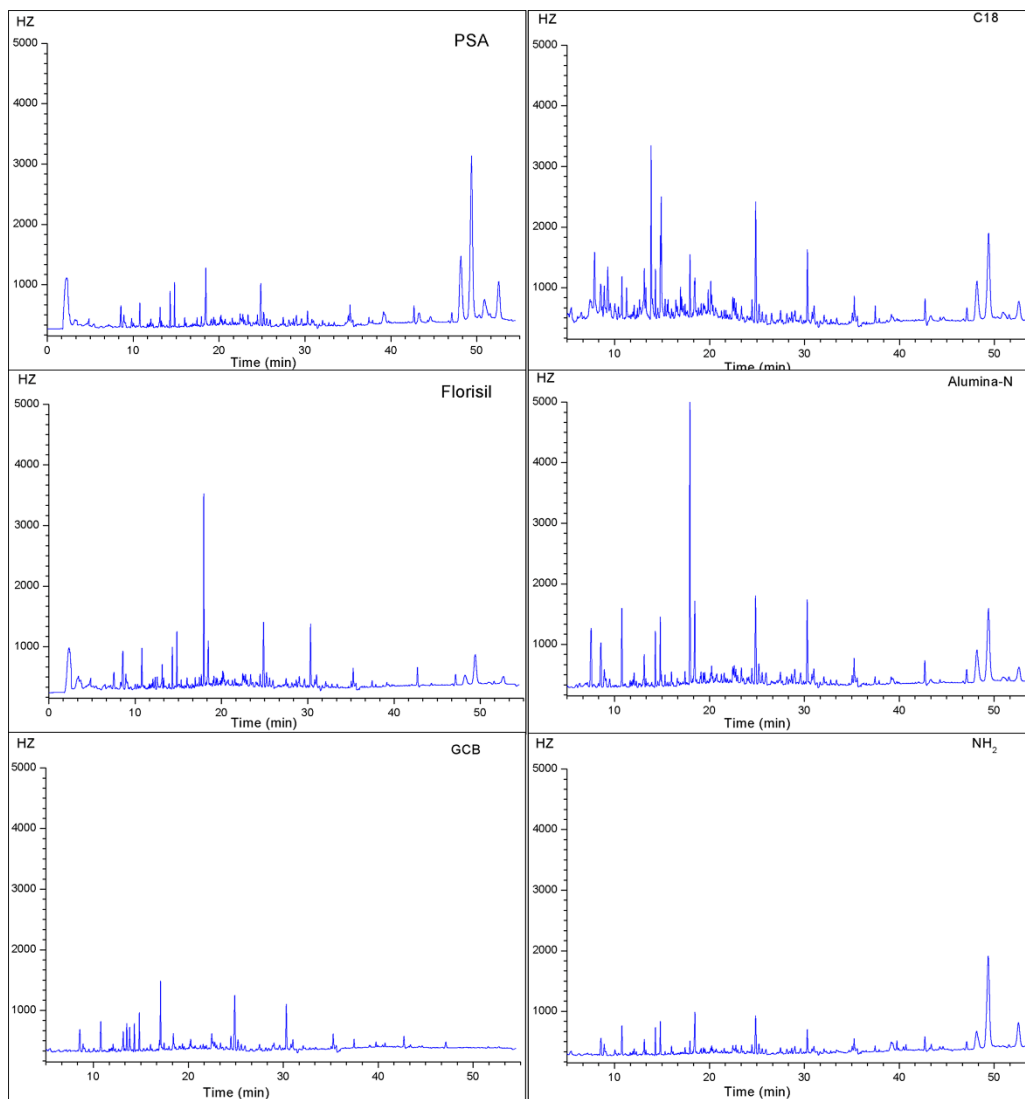


Figure S2. GC-ECD chromatogram of negative dried tangerine peel extract purified by various sorbents including Cleanert PSA, C₁₈, Florisil, Alumina N, PestiCarb (GCB) and NH₂.

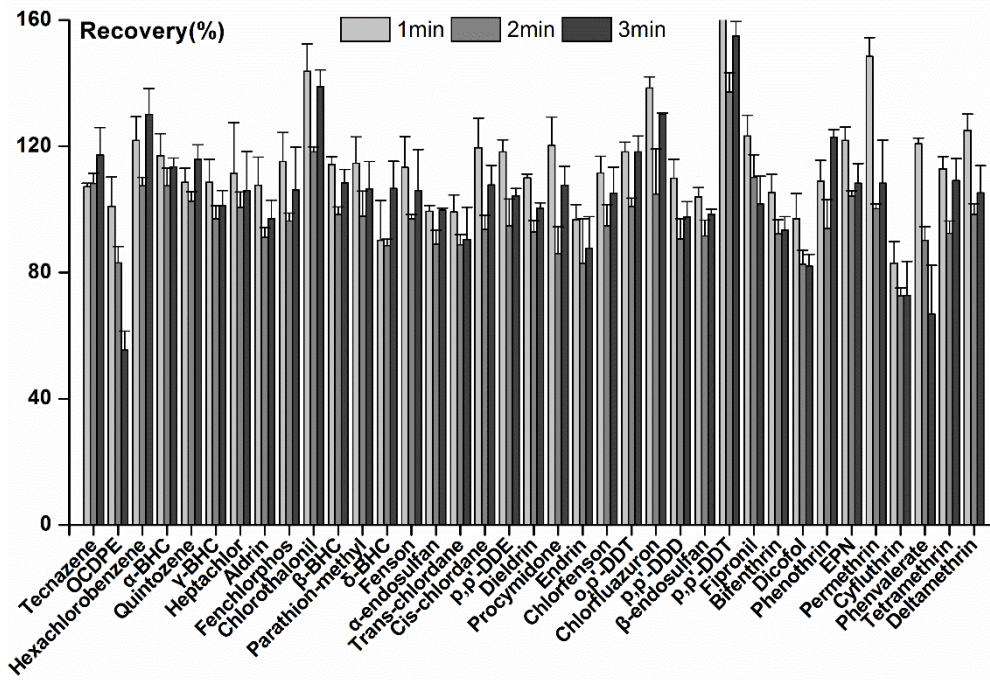


Figure S3. Recovery rates of spiked tangerine peel solution at a concentration of 0.05 mg kg^{-1} ($n = 3$) under various operating time of 1, 2 and 3 min.

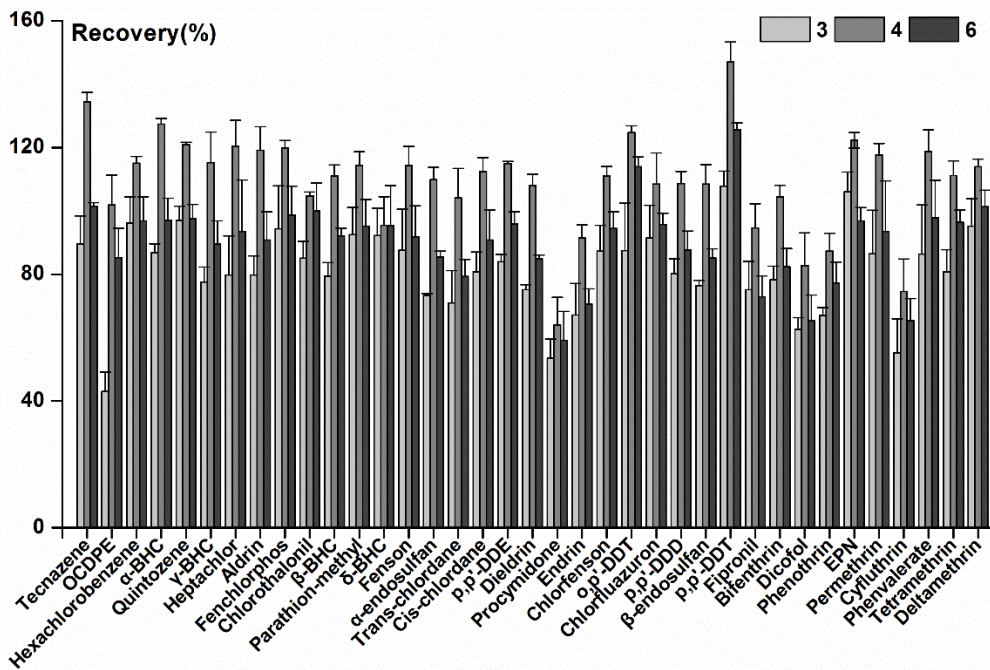


Figure S4. Recovery rates of spiked tangerine peel solution at a concentration of 0.05 mg kg^{-1} ($n = 3$) under 3, 4 and 6 times of push-pull cycle.

Table S1. Precision, accuracy (represented by RE) and repeatability of 37 pesticides

Pesticide	Precision (RSD) / %		0.025 mg kg ⁻¹		0.05 mg kg ⁻¹		0.5 mg kg ⁻¹	
	Intra-day	Inter-day	RE / %	RSD / %	RE / %	RSD / %	RE / %	RSD / %
Tecnazene	0.48	6.02	91.7	12.9	100.7	8.3	94.2	3.2
OCDPE	0.15	7.86	128.6	8.0	105.7	11.7	98.6	5.2
Hexachlorobenzene	0.95	3.95	65.6	3.7	70.9	8.1	71.0	2.7
α -BHC	0.07	4.27	102.2	5.1	112.2	5.8	102.9	5.5
Quintozene	0.02	4.43	77.3	11.1	86.7	7.6	80.1	3.1
γ -BHC	0.62	4.24	90.1	8.1	101.2	12.4	100.0	4.1
Heptachlor	0.40	11.3	107.9	12.4	119.8	7.4	113.5	4.9
Aldrin	0.21	3.38	112.2	15.6	120.8	10.1	112.8	3.1
Fenchlorphos	2.43	1.70	80.3	4.4	91.9	9.3	91.4	2.6
Chlorothalonil	8.81	8.23	73.2	7.3	87.3	12.0	84.0	1.5
β -BHC	3.43	3.30	82.1	4.3	95.0	8.4	104.2	2.4
Parathion-methyl	4.38	2.96	76.5	7.0	85.8	8.7	83.7	8.0
δ -BHC	2.82	3.26	91.4	19.1	109.2	8.3	97.8	2.0
Fenson	3.59	3.43	106.4	13.1	115.5	9.7	110.4	1.4
α -Endosulfan	1.66	2.25	103.2	17.8	109.4	10.8	106.7	4.3
<i>trans</i> -Chlordane	0.27	3.44	93.2	7.8	110.1	10.7	105.2	3.3
<i>cis</i> -Chlordane	0.32	6.11	91.5	7.9	108.6	9.5	110.2	4.6
p,p'-DDE	1.17	3.87	101.7	13.1	112.5	9.0	105.8	15.6
Dieldrin	0.56	5.21	84.4	2.0	90.8	8.2	95.2	13.6
Procymidone	3.20	9.12	92.6	7.0	108.4	9.3	106.8	16.5
Endrin	0.41	6.96	87.3	8.3	95.6	8.3	93.0	14.1
Chlorfenson	4.93	2.52	102.0	14.1	116.5	3.6	113.8	6.8
o,p'-DDT	4.26	11.9	66.8	5.7	61.6	4.8	70.6	2.6
Chlorfluazuron	2.60	9.90	94.3	10.5	108.8	9.0	104.1	14.3
p,p'-DDD	2.10	6.92	91.0	11.3	103.0	7.3	97.5	6.4
β -Endosulfan	0.47	3.09	97.1	9.0	110.0	7.2	117.7	5.1
p,p'-DDT	8.11	10.8	74.8	1.4	85.2	12.6	77.4	6.1
Fipronil	4.82	5.89	97.1	11.5	110.4	10.2	104.9	7.1
Bifenthrin	1.28	3.95	75.7	7.1	84.8	7.8	107.9	14.5
Dicofol	5.17	9.11	78.2	7.3	87.4	9.7	85.9	11.5
Phenothrin	2.93	1.31	94.1	16.1	110.5	7.3	109.2	9.0
EPN	2.09	6.44	88.9	1.0	104.0	10.9	97.5	1.7
Permethrin	4.54	2.71	106.8	5.5	90.9	12.7	96.8	1.5
Cyfluthrin	1.93	6.16	79.4	13.5	97.8	6.3	95.6	2.6
Fenvalerate	9.59	7.37	76.6	6.4	105.0	7.0	116.4	4.3
Tetramethrin	1.85	3.43	91.1	5.6	62.4	9.8	81.1	3.9
Deltamethrin	1.45	4.73	82.4	5.7	91.4	6.4	92.2	3.4

OCDPE: octachlorodipropyl ether; BHC: benzene hexachloride; DDE: dichlorodiphenyldichloroethylene; DDT: dichlorodiphenyltrichloroethane; DDD: dichlorodiphenyldichloroethane; EPN: *O*-ethyl *O*-(4-nitrophenyl) phenylphosphonothioate.

Table S2. The residue level of pesticides in positive dried tangerine peels

Sample No.	Hexachlorobenzene / (mg kg ⁻¹)	p,p'-DDE / (mg kg ⁻¹)	Procymidone / (mg kg ⁻¹)	Dicofol / (mg kg ⁻¹)
4	0.07	0.04	— ^a	0.2
7	0.1	— ^a	— ^a	0.7
2-1	0.2	— ^a	0.4	— ^a
3-1	— ^a	— ^a	— ^a	0.2
4-1	0.03	— ^a	— ^a	— ^a
W-8	0.08	— ^a	— ^a	0.6
S-5	0.05	0.1	0.8	0.9
T-3	— ^a	— ^a	— ^a	0.6

^aNot detected. DDE: dichlorodiphenyldichloroethylene.