

Supplementary Material

Exploration of modified pretreatment process coupled with GC-MS/MS for determination of 18 phthalates in edible oils

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See DOI: 10.1039/x0xx00000x

Table S1. Retention time (RT), Quantification ion, and Identification ion for 18 PAEs

PAEs	RT (min)	Quantification Ion pair	CE (eV)	Identification ion (1)	CE (eV)	Identification ion (2)	CE (eV)
DMP	7.545	163.0>77.0	25	163.0>135.0	15	163.0>92.0	25
DEP	8.420	149.0>65.0	25	149.0>93.0	15	177.0>149.0	10
DAP	9.298	149.0>65.0	25	149.0>93.0	15	149.0>121.0	10
DIBP	10.057	149.0>65.0	25	149.0>93.0	20	149.0>121.0	15
DBP	10.758	149.0>65.0	25	149.0>93.0	20	149.0>121.0	15
DMEP	11.083	149.0>65.0	25	149.0>93.0	15	149.0>121.0	10
BMPP	11.786	149.0>65.0	25	149.0>93.0	20	149.0>121.0	15
DEEP	12.118	149.0>65.0	25	149.0>93.0	15	149.0>121.0	10
DPP	12.456	149.0>65.0	25	149.0>93.0	20	149.0>121.0	10
DHXP	14.552	149.0>65.0	25	149.0>93.0	15	149.0>121.0	15
BBP	14.663	149.0>65.0	25	149.0>93.0	15	149.0>121.0	10
DBEP	16.100	149.0>65.0	25	149.0>93.0	15	149.0>121.0	10
DCHP	16.710	149.0>65.0	25	149.0>93.0	20	149.0>121.0	15
DEHP	16.967	149.0>65.0	25	149.0>93.0	15	149.0>121.0	10
DPHP	17.097	224.0>77.0	25	224.0>141.0	20	224.0>197.0	10
DNOP	19.218	149.0>65.0	25	149.0>93.0	20	149.0>121.0	15
DINP	20.006	149.0>65.0	25	149.0>93.0	20	149.0>121.0	15
DNP	21.151	149.0>65.0	25	149.0>93.0	20	149.0>121.0	15

Table S2. Matrix effect of 18 PAEs

PAEs	Regression equation of standard solution(A)	R ²	Regression equation of matrix solution(B)	R ²	ME (%)
DMP	$y = 786320.64 x + 2921.77$	0.9998	$y = 725002.82 x - 9686.73$	0.9999	-7.80
DEP	$y = 834615.96 x + 6529.19$	0.9999	$y = 819673.81x - 14141.01$	0.9998	-1.79
DAP	$y = 348722.27 x + 2691.01$	0.9992	$y = 337743.41 x - 5619.82$	0.9999	-3.15
DIBP	$y = 1260105.25 x + 27025.42$	0.9994	$y = 1210159.83 x - 10677.70$	0.9999	-3.96
DBP	$y = 1478960.39 x + 8259.17$	0.9999	$y = 1466439.72 x + 122414.04$	0.9998	-0.85
DMEP	$y = 86698.56 x + 410.45$	0.9998	$y = 82038.81 x - 1072.60$	0.9996	-5.37
BMPP	$y = 775722.19 x + 4733.04$	0.9999	$y = 754763.44 x - 19074.39$	0.9993	-2.70
DEEP	$y = 193102.06 x + 1172.05$	0.9997	$y = 202688.65 x - 4515.84$	0.9998	4.96
DPP	$y = 1340529.23 x + 6969.42$	0.9998	$y = 1327752.81 x - 22942.31$	0.9999	-0.95
DHXP	$y = 1409269.02 x - 12353.93$	0.9998	$y = 1395979.59 x - 28125.77$	0.9999	-0.17
BBP	$y = 571052.38 x - 10219.48$	0.9998	$y = 554732.14 x - 12075.49$	0.9999	-2.86
DBEP	$y = 505971.77 x - 4297.72$	0.9990	$y = 536855.57 x - 9444.53$	0.9999	6.10
DCHP	$y = 1854453.13 x + 31451.18$	0.9998	$y = 1756190.64 x - 42217.38$	0.9996	-5.30
DEHP	$y = 1308150.49 x + 32923.31$	0.9996	$y = 12868333.17 x + 248259.86$	0.9998	-1.63
DPHP	$y = 443692.33 x + 3177.21$	0.9997	$y = 424471.88 x - 8629.38$	0.9998	-4.33
DNOP	$y = 2092497.15x + 16700.15$	0.9988	$y = 1897734.88 x - 38304.89$	0.9999	-9.31
DINP	$y = 18950.31 x + 1416.79$	0.9990	$y = 18600.72 x + 569.98$	0.9989	-1.84
DNP	$y = 593548.69 x + 22314.49$	1.0000	$y = 571509.75 x - 2543.06$	1.0000	-3.71

Table S3. Determination of 18 PAEs in edible vegetable oils (n=6, mg·kg⁻¹)

PAEs	Colza oils	Soybean oils	Peanut oils	Blend oil	Olive oil
DMP	Nd	Nd	Nd	Nd	Nd
DEP	Nd	Nd	Nd	Nd	Nd
DAP	Nd	Nd	Nd	Nd	Nd
DIBP	Nd	Nd	Nd	Nd	Nd
DBP	0.106–0.484	0.135–0.391	Nd	Nd	1.15
DMEP	Nd	Nd	Nd	Nd	Nd
BMPP	Nd	Nd	Nd	Nd	Nd
DEEP	Nd	Nd	Nd	Nd	Nd
DPP	Nd	Nd	Nd	Nd	Nd
DHXP	Nd	Nd	Nd	Nd	Nd
BBP	Nd	Nd	Nd	Nd	Nd
DBEP	Nd	Nd	Nd	Nd	Nd
DCHP	Nd	Nd	Nd	Nd	Nd
DEHP	0.150	Nd	0.106	Nd	Nd
DPHP	Nd	Nd	Nd	Nd	Nd
DNOP	Nd	Nd	Nd	Nd	Nd
DINP	Nd	Nd	Nd	Nd	Nd
DNP	Nd	Nd	Nd	Nd	Nd

Nd, not detected, <LOQ.

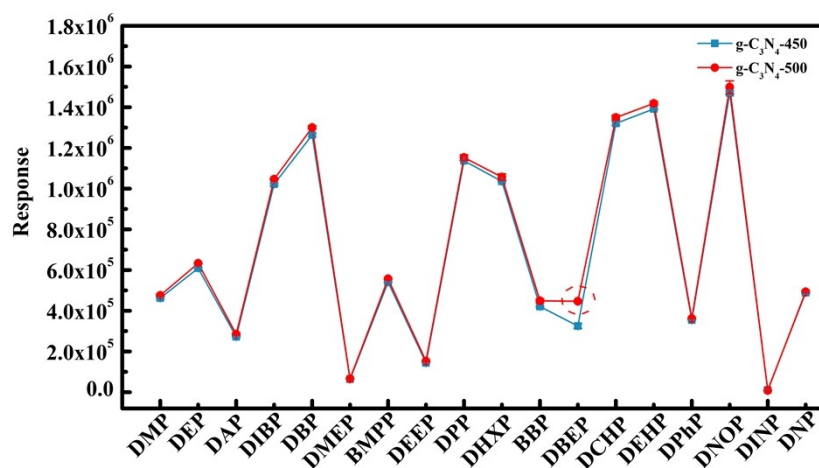


Fig S1. Comparison of the clean-up effect of g-C₃N₄ synthesized under different pyrolysis temperature (the volume ratio of methanol and ethyl hexanoate, extraction temperature, extraction time, the amount of g-C₃N₄, PSA and the spiked concentration of 18 PAEs are 4000:400, 28°C, 12 min, 30 mg ,15mg and 3.2 mg·kg⁻¹ respectively)

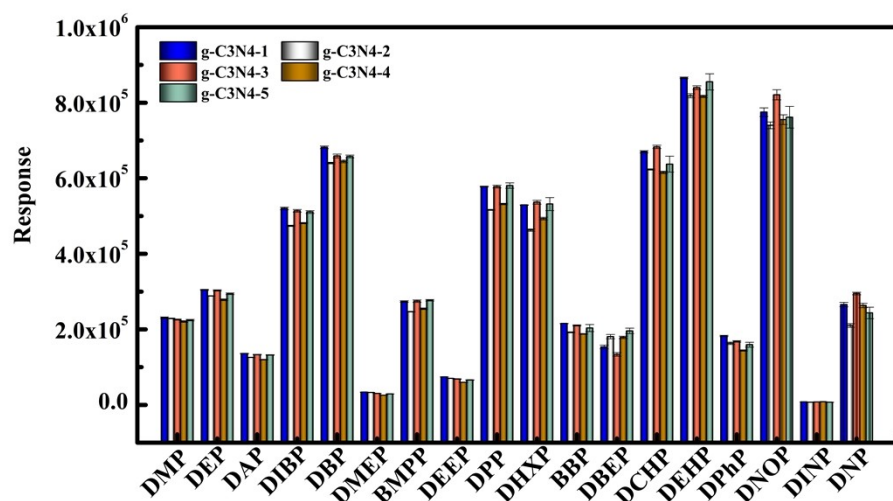


Figure S2. Comparison of the signal response of inter-batch synthesized g-C₃N₄ (the volume ratio of methanol and ethyl hexanoate, extraction temperature, extraction time, the amount of g-C₃N₄, PSA and the spiked concentration of 18 PAEs are 4000:400, 28°C, 12 min, 30 mg ,15mg and 1.6 mg·kg⁻¹ respectively)

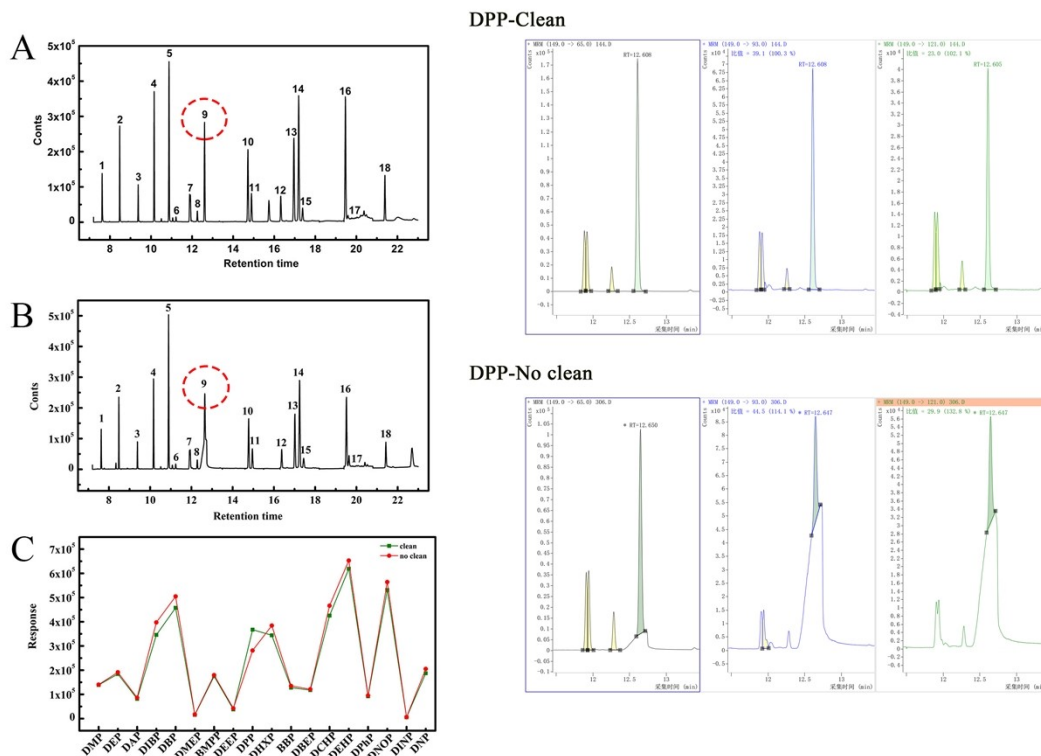


Figure S3 (A). the TIC chromatogram of matrix addition of PAEs after clean-up by g-C₃N₄ /PSA mixed absorbent acquired in MRM mode (black canola oil); (B). the TIC chromatogram of matrix addition of PAEs without clean-up process acquired in MRM mode (black canola oil); (C). Comparison of the signal response after clean-up and without clean-up process (the volume ratio of methanol and ethyl hexanoate, extraction temperature, extraction time, the amount of g-C₃N₄, PSA and the spiked concentration of 18 PAEs are 2000:200, 28°C, 12 min, 30 mg, 15mg, and 1.2 mg·kg⁻¹ respectively)
(1. DMP, 2. DEP, 3. DAP, 4. DIBP, 5. DBP, 6. DMEP, 7. BMPP, 8. DEEP, 9. DPP, 10. DHXP, 11. BBP, 12. DBEP, 13. DCHP, 14. DEHP, 15 DPHP, 16, DNOP, 17 DINP, 18. DNP)

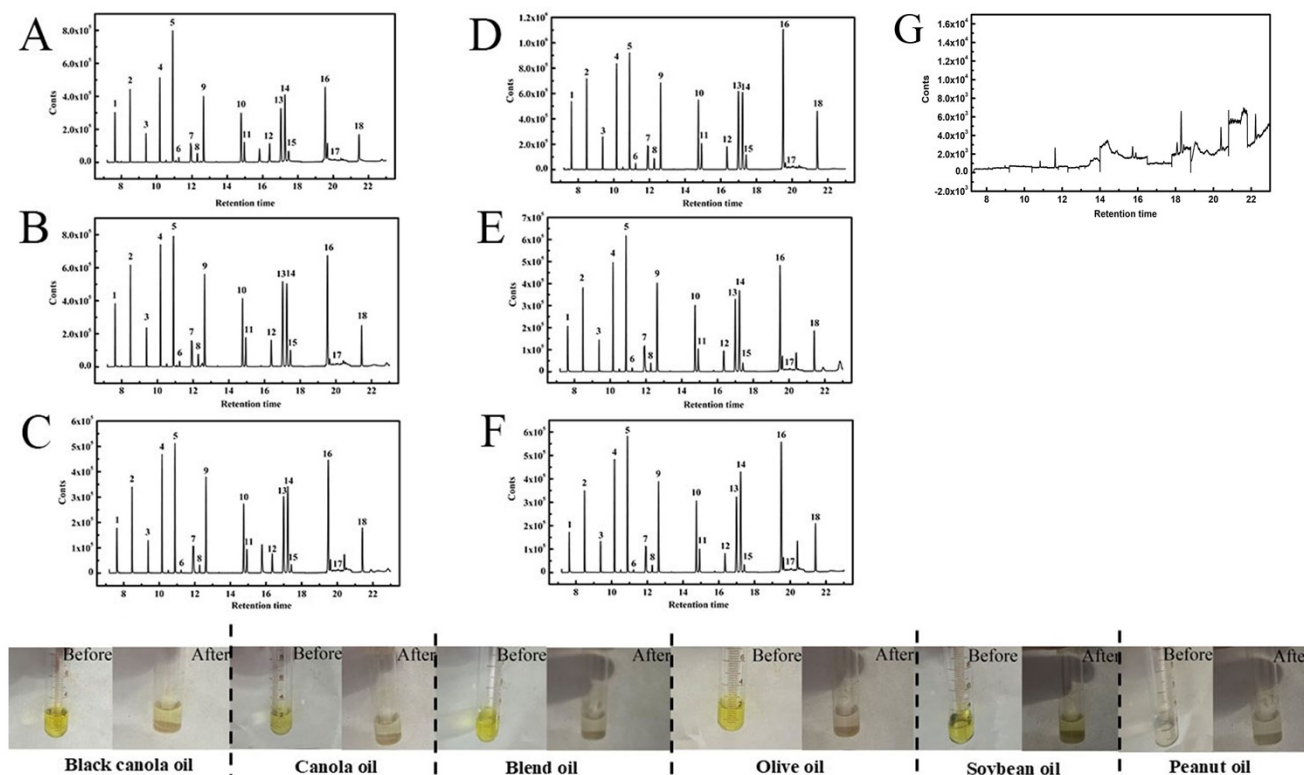
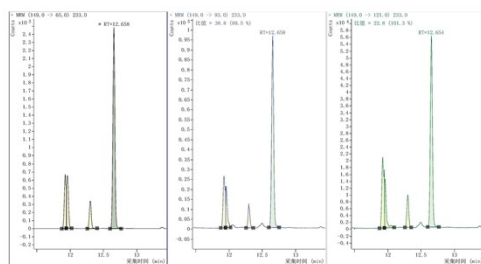


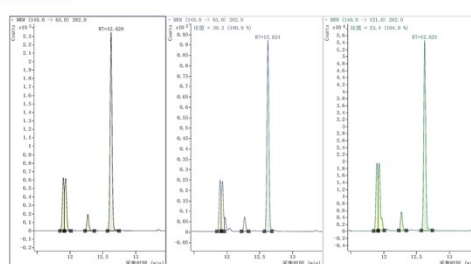
Figure S4. The TIC chromatography of 18 PAEs added in different matrix-solution after clean-up by $g\text{-C}_3\text{N}_4/\text{PSA}$ mixed absorbent acquired in MRM mode (A. black canola oil; B. yellow canola oil; C. blend oil; D. olive oil; E. soybean oil; F. peanut oil). (G) the TIC Chromatography of blank solvent acquired in MRM mode.

(1. DMP, 2. DEP, 3. DAP, 4. DIBP, 5. DBP, 6. DMEP, 7. BMPP, 8. DEEP, 9. DPP, 10. DHXP, 11. BBP, 12. DBEP, 13. DCHP, 14. DEHP, 15. DPHP, 16. DNOP, 17. DINP, 18. DNP)

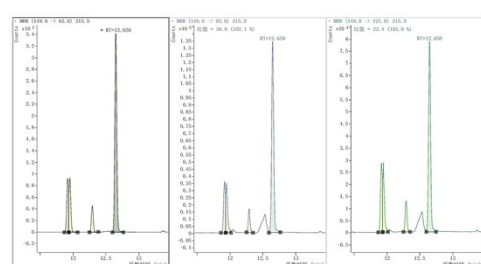
DPP-black canola oil



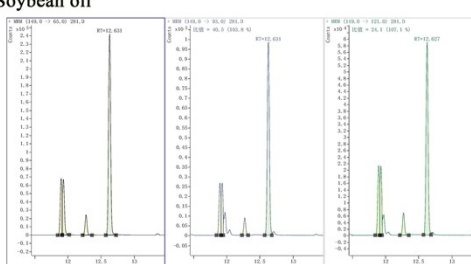
DPP-Olive oil



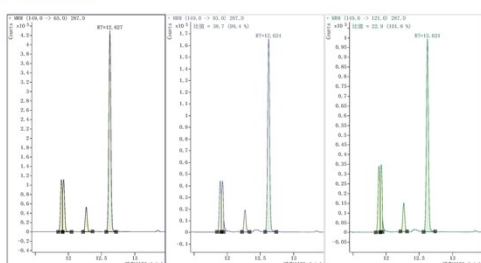
DPP-canola oil



DPP-Soybean oil



DPP-blend oil



DPP-peanut oil

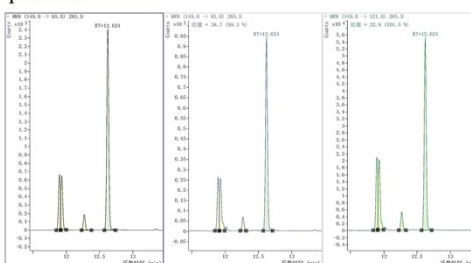


Figure S5. The relative MRM chromatography of DPP of different matrix-solution after clean-up by $g\text{-C}_3\text{N}_4/\text{PSA}$ mixed absorbent

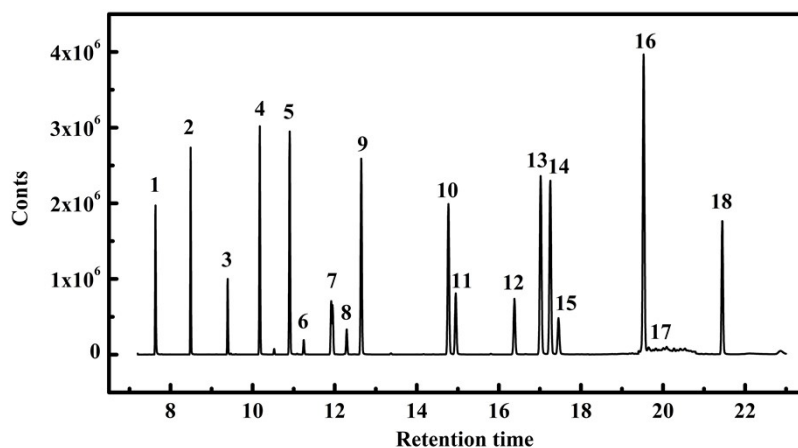


Figure S6. (A) the TIC chromatogram of matrix-matched standard solution of 18 PAEs acquired in MRM mode (black canola oil matrix, the volume ratio of methanol and ethyl hexanoate, extraction temperature, extraction time, the amount of g-C₃N₄, PSA, and the spiked concentration of 18 PAEs are 4000:400, 28°C, 12 min, 30 mg, 15 mg and 8.0 mg·kg⁻¹ respectively)

(1. DMP, 2. DEP, 3. DAP, 4. DIBP, 5. DBP, 6. DMEP, 7. BMPP, 8. DEEP, 9. DPP, 10. DHXP, 11. BBP, 12. DBEP, 13. DCHP, 14. DEHP, 15. DPHP, 16. DNOP, 17. DINP, 18. DNP)