Supplementary Data for

Efficient degradation of 4-acetamidoantipyrin by thermally activated persulfate system

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List of Supplementary Data

Text S1 Separation conditions and operating parameters for UPLC-Orbitrap-MS

Separation conditions for UPLC: the mobile phase comprised of water with 0.1% acetic acid (phase A) and methanol with 0.1% acetic acid (phase B) at a flow rate of 0.3 mL/min. The gradient program was: 0 min, 95% A and 5% B; 4 min, 60% A and 40% B; 5 min, 100% B; 6.5-8 min, 95% A and 5% B. Column temperature was 40 ℃ and the volume of sample was 10 μL.

Operating parameters of MS: mass spectra scan range 100-600 Da, capillary voltage 3500 V, source temperature 110 ºC, dissolvation temperature 350 ℃, cone hole gas flow 50 L/h, desolvent gas flow 800 L/h.

Figure S1

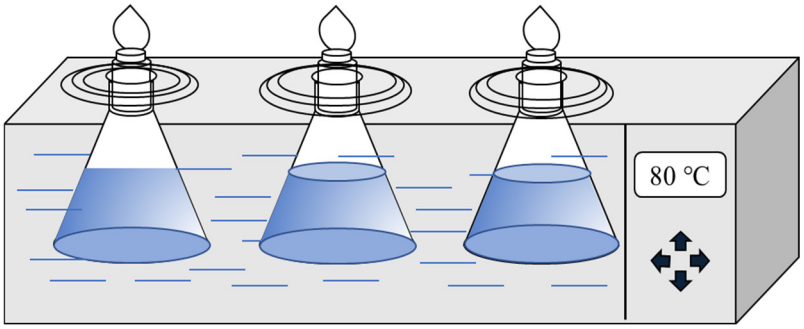


Figure S1. Schematic of experimental reactors.

Figure S2

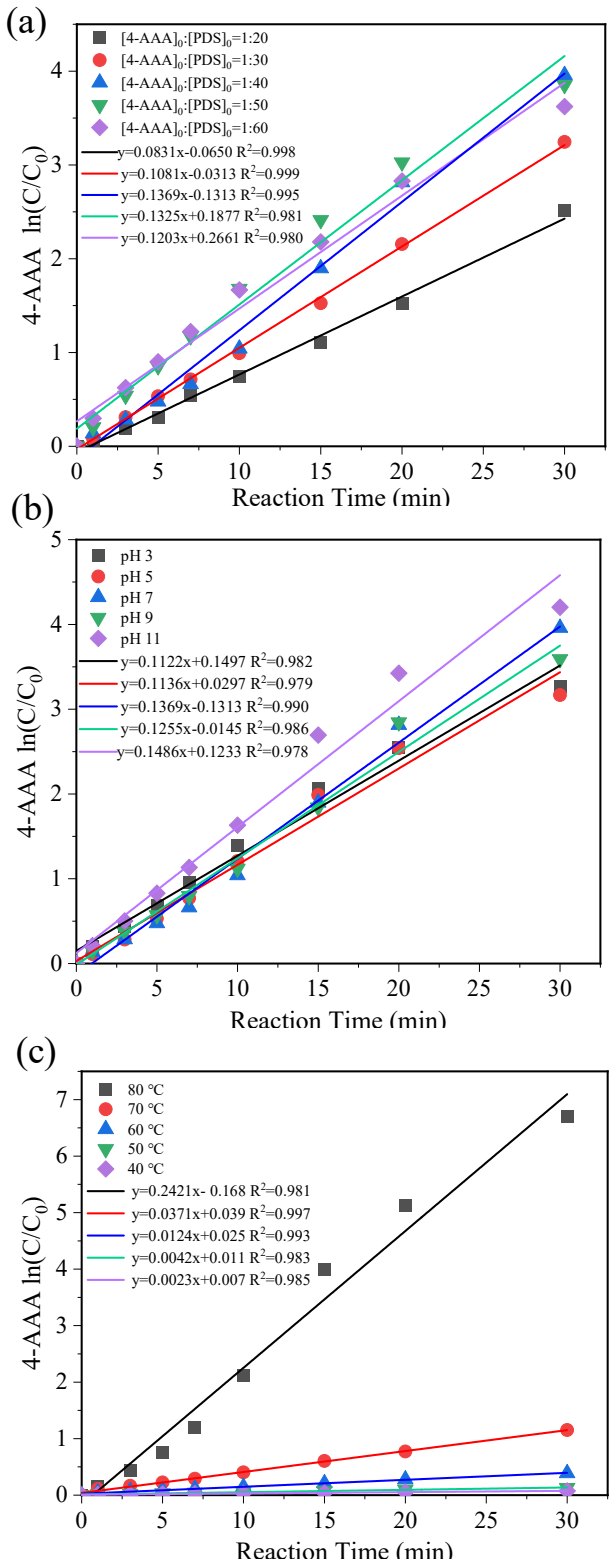


Figure S2. Pseudo first order reaction kinetic fitting results for 4-AAA degradation with different PDS concentrations (a), initial pH (b) and temperature (c) in thermally activated PDS systems. Experimental conditions: $[4\text{-AAA}]_0 = 40.77$ μM, $[PDS]_0 = 1.63$ mM, $pH_0 = 7$, T = 80 °C.

Figure S3

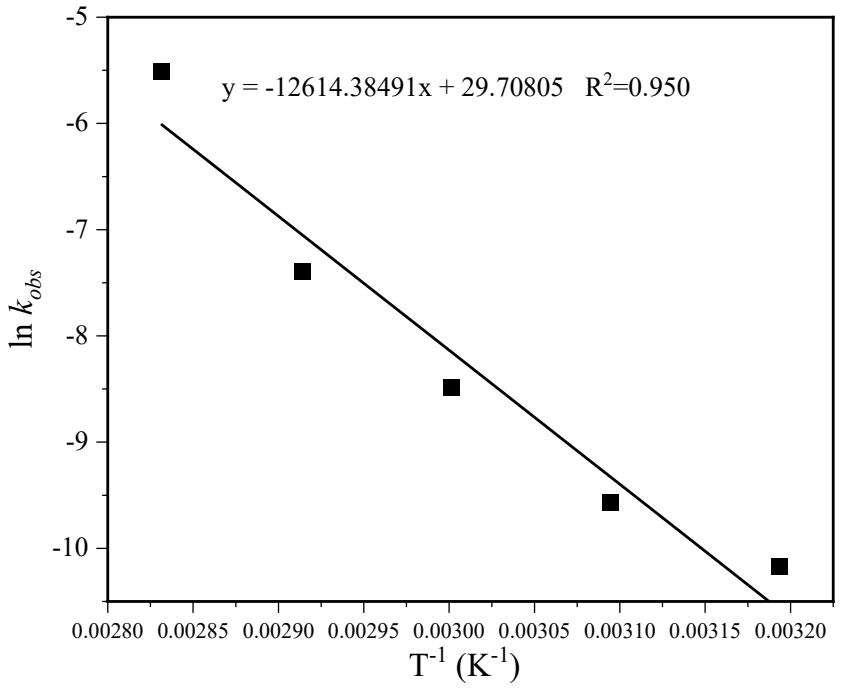


Figure S3. Calculation of E_a of 4-AAA in thermally activated PDS systems. Experimental conditions: $[4\text{-AAA}]_0$ = 40.77 μM, $[PDS]_0$ = 1.63 mM, pH_0 = 7.

Notes: Based on Arrhenius equation $\ln k = -\frac{E}{RT} + \ln A$, the slope of plot ln$k$ vs T^{-1} in Figure S2 (i.e., -12614.38491) equals $-\frac{E}{R}$. As the value of R was 8.314 J/(mol·K), the value of E_a was calculated to be 104.88 kJ/mol.

Figure S4

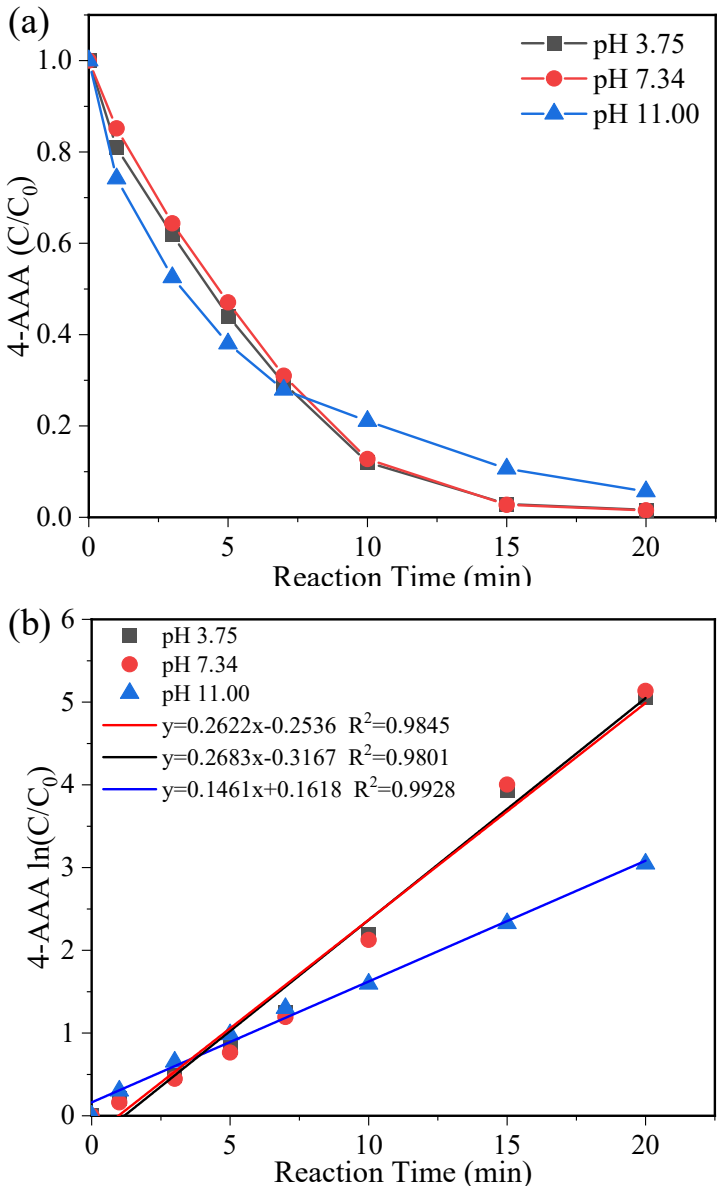
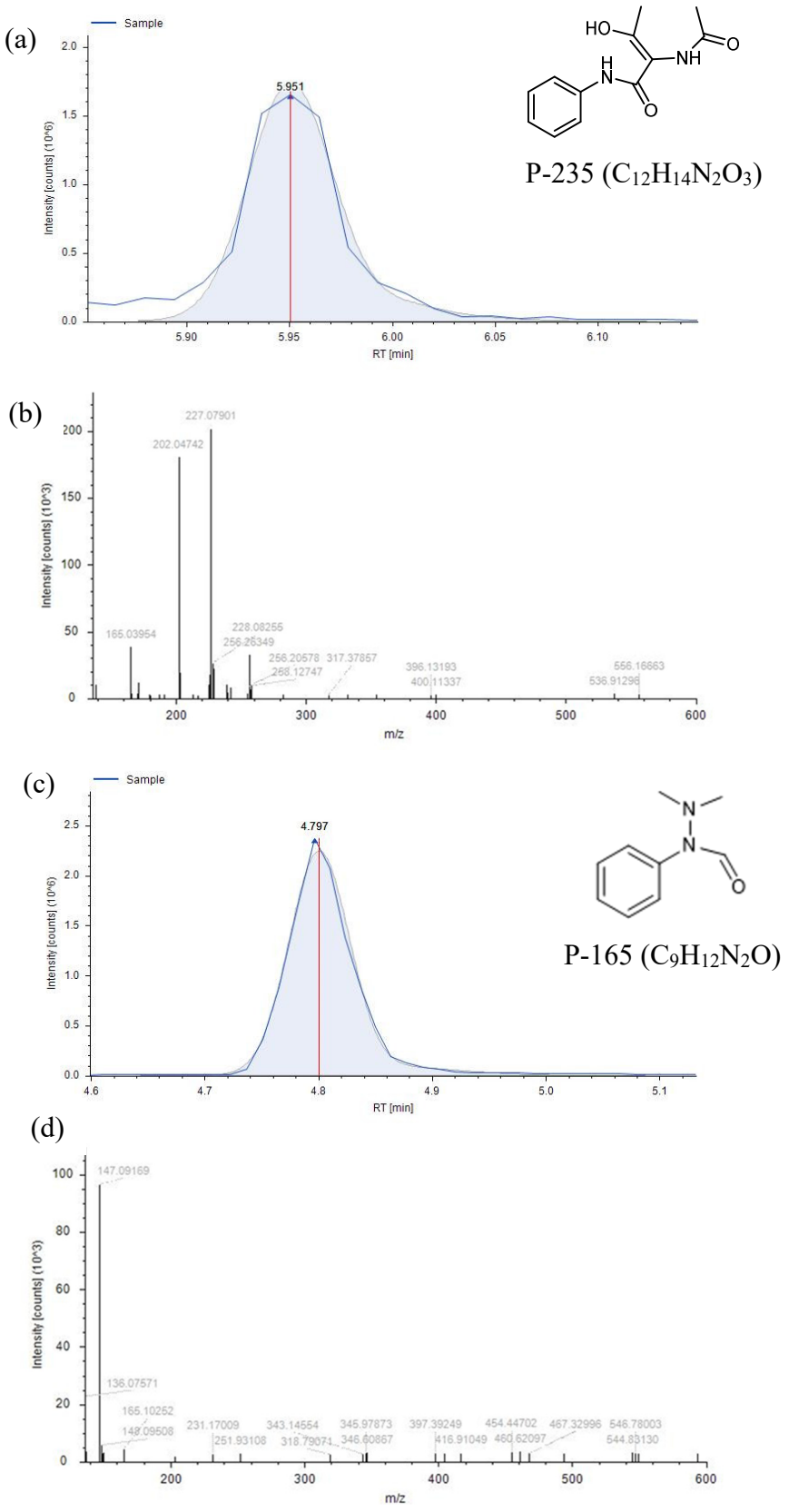


Figure S4. Validation experiments of the optimal conditions by RSM model. Experimental conditions: $[4\text{-AAA}]_0$ = 40.77 μM, $[PDS]_0$ = 2.88 mM, pH_0 = 3.75, T = 79.24 ℃ (a), $[4\text{-AAA}]_0$ = 40.77 μM, $[PDS]_0$ = 1.92 mM, pH_0 = 7.34, T = 79.53 ℃ (b), $[4\text{-AAA}]_0$ = 40.77 μM, $[PDS]_0$ = 2.36 mM, pH_0 = 11.00, T = 79.11 ℃ (c).

Figure S5


(a)
Sample
5.951
Intensity [counts] (10^6)
RT [min]
HO
NH
H
N
O
P-235 ($C_{12}H_{14}N_2O_3$)
(b)
227.07901
202.04742
165.03954
228.08255
256.26349
256.20578
317.37857
268.12747
396.13193
400.11337
556.16863
536.91296
Intensity [counts] (10^3)
m/z
(c)
Sample
4.797
Intensity [counts] (10^6)
RT [min]
N
N
O
P-165 ($C_9H_{12}N_2O$)
(d)
147.09169
136.07571
165.10252
148.09508
231.17009
251.93108
343.14554
318.79071
345.97873
346.60867
397.39249
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546.78003
544.83130
Intensity [counts] (10^3)
m/z

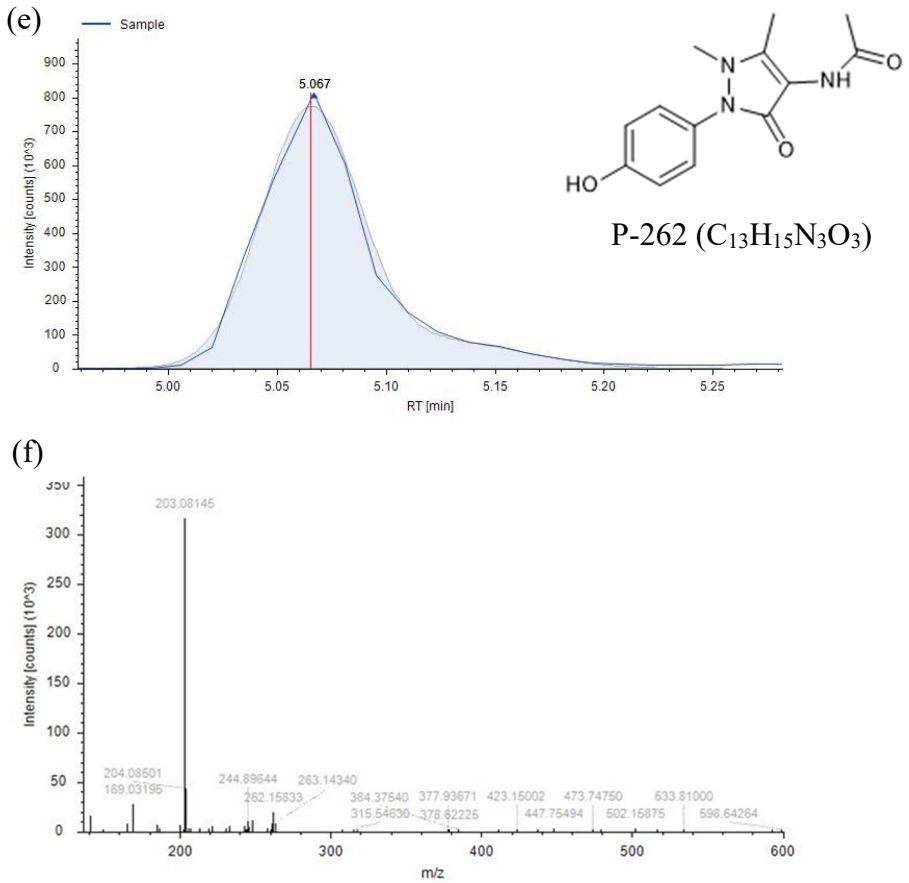

Figure S5. Chromatography and mass spectrometry of reaction products and structural formula.

Figure S6

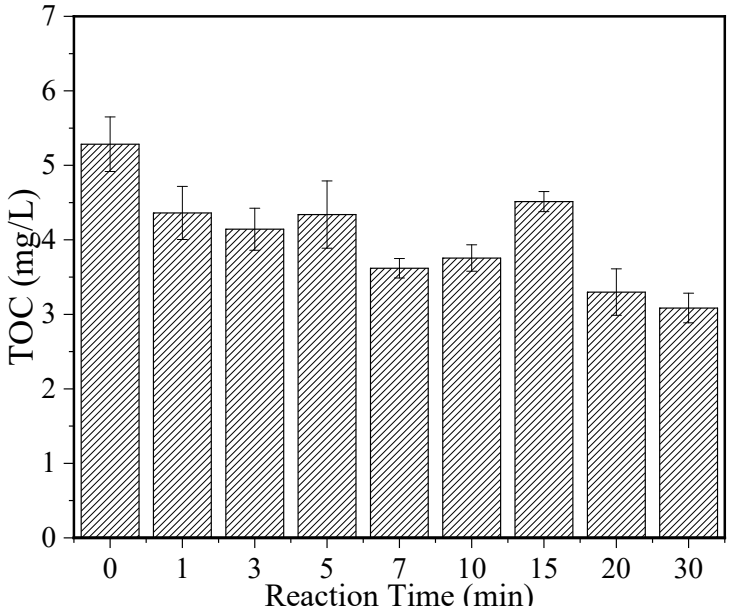

Figure S6. TOC changes in degradation of 4-AAA by thermal/PS system. Experimental conditions: $[4\text{-AAA}]_0 = 40.77$ μM, $[PDS]_0 = 1.63$ mM, $pH_0 = 7.00$, T = 80 ℃.