Electronic Supplementary Material (ESI) for Analytical Methods.

Electronic Supplementary Information

Monolith stationary phases prepared via cyclic anhydride ring-opening polymerization as tunable platforms for chromatographic applications

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Summary

This electronic supplementary material file includes additional results and information as described in the text of the main article, including:

Figures S1–S5.

Tables S1 & S2.

References related the comparison study.

Fig. S1

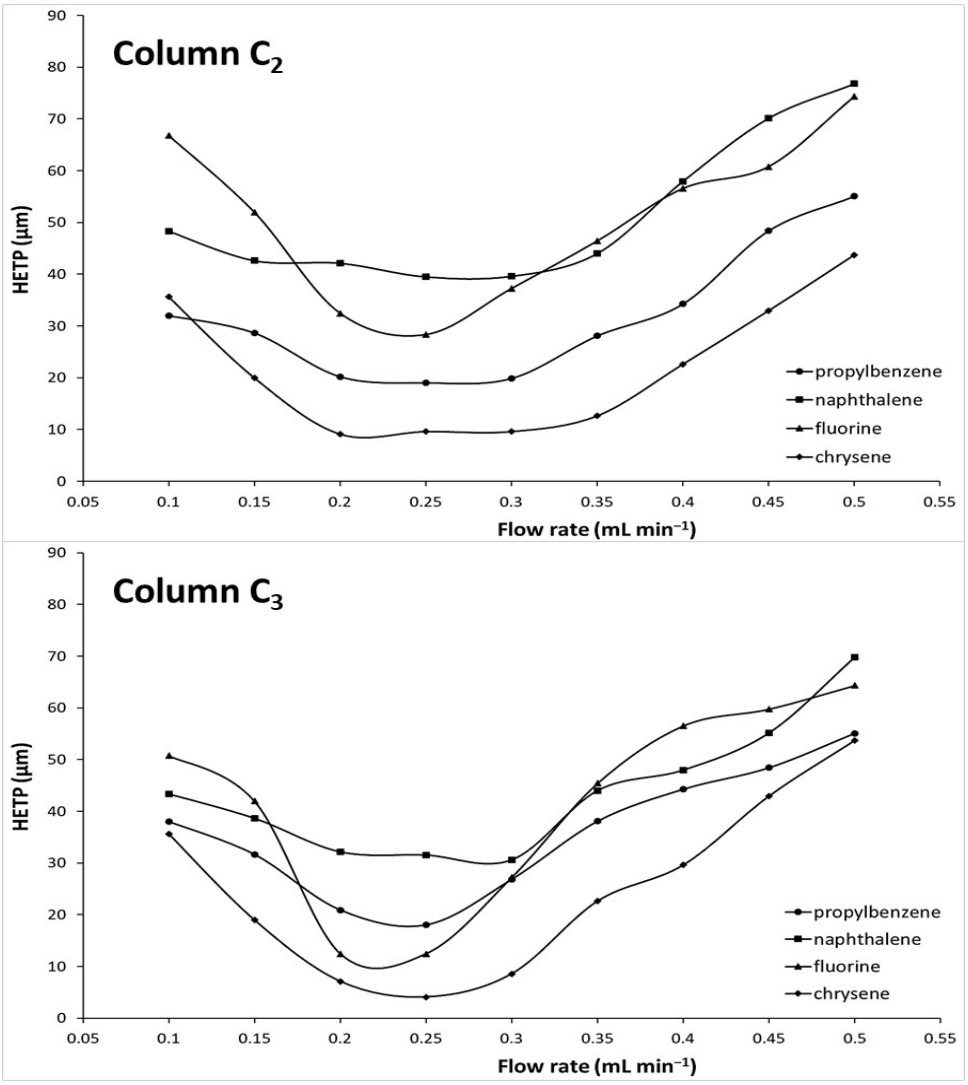

Fig. S1 Van Deemter curves for some of the studied compounds injected in C_2 and C_3 columns.

Table S1

Table S1 Repeatability (in terms of run-to-run and day-to-day) and reproducibility (in terms of column-to-column) studies for the prepared columns.

	Column	C_2 column					C_3 column				
	Solute	butylbenzene		pentylbenzene			butylbenzene		pentylbenzene		
		t_R	H	t_R	H	R_s	t_R	H	t_R	H	R_s
Run-to-run	1	3.31	20.75	4.12	23.28	2.36	4.23	18.93	5.55	15.32	3.94
	2	3.28	22.30	4.11	23.76	2.53	4.22	16.85	5.56	17.91	3.81
	3	3.29	22.94	4.13	26.07	2.66	4.22	17.93	5.55	17.47	3.69
	4	3.29	18.47	4.11	26.88	2.48	4.23	20.81	5.53	15.50	3.94
	5	3.28	19.07	4.10	25.13	2.43	4.23	19.78	5.53	15.02	3.92
	Avg.	3.29	20.71	4.11	25.02	2.49	4.23	18.86	5.54	16.24	3.86
	%RSD	0.37	9.42	0.28	6.06	4.53	0.13	8.20	0.24	8.25	2.83
Day-to-day	1	3.29	20.71	4.11	25.02	2.49	4.23	18.86	5.54	16.24	3.86
	2	3.27	18.63	4.13	26.76	2.50	4.23	15.75	5.58	15.72	3.92
	3	3.27	21.40	4.09	26.07	2.82	4.24	16.84	5.57	18.33	3.81
	4	3.26	18.05	4.13	23.88	2.65	4.21	18.15	5.55	18.04	3.98
	5	3.25	22.66	4.12	23.13	2.71	4.21	18.62	5.54	19.28	3.91
	Avg.	3.27	20.29	4.12	24.97	2.63	4.22	17.64	5.56	17.52	3.90
	%RSD	0.45	9.48	0.41	6.00	5.35	0.32	7.46	0.33	8.51	1.65
Column-to-column	1	3.27	20.29	4.12	24.97	2.63	4.23	17.64	5.56	17.52	3.90
	2	3.88	31.04	4.81	32.33	2.28	4.61	27.73	6.12	29.01	3.97
	3	2.93	24.33	3.85	27.01	2.58	3.84	24.70	5.03	24.90	3.32
	4	3.49	28.14	4.07	33.82	2.51	4.06	22.43	5.27	21.65	3.56
	Avg.	3.41	25.98	4.24	29.53	2.50	4.19	23.13	5.50	23.27	3.69
	%RSD	11.14	18.11	10.94	14.28	6.58	7.77	18.39	8.55	20.95	8.23

t_R: retention time (min), H: height equivalent to theoretical plates (μm), and R_s: resolution factor.

Fig. S2

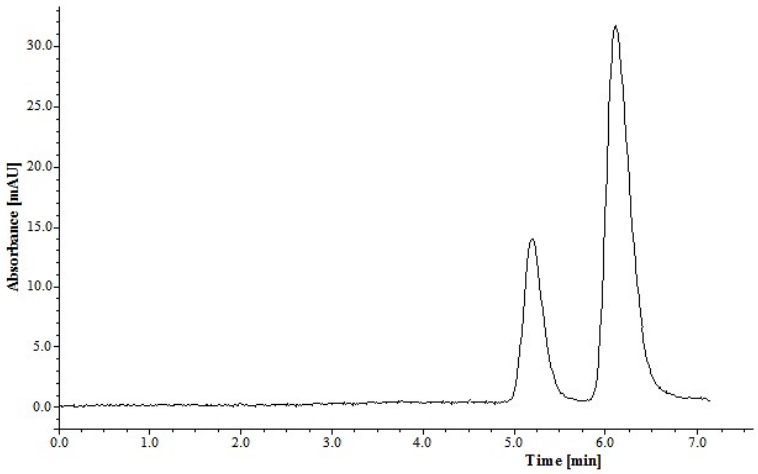

Fig. S2 Separation chromatogram of caffeine and ibuprofen extracted from Profinal-XP tablets using C_3 column. Conditions: mobile phase: acetonitrile/water with 0.1% formic acid (60/40, v/v), flow rate: 0.30 mL min^{-1}, detection wavelength: UV at 215 nm, column temperature: 30^{o}C. Peaks identification by order of elution: 1- caffeine and 2- ibuprofen.

Fig. S3

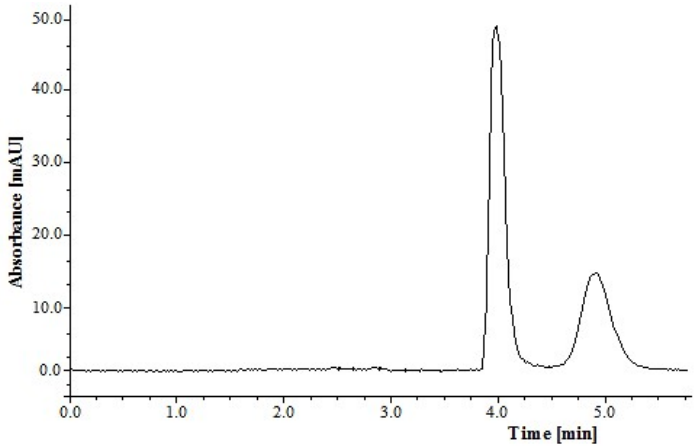

Fig. S3 Separation chromatogram of paracetamol and chlorzoxazone extracted from Relaxon capsules using C_3 column. Conditions: mobile phase: acetonitrile/water with 0.1% formic acid (55/45, v/v), flow rate: 0.25 mL min^{-1}, detection wavelength: UV at 270 nm, column temperature: 30^{o}C. Peaks identification by order of elution: 1- paracetamol and 2- chlorzoxazone.

Fig. S4

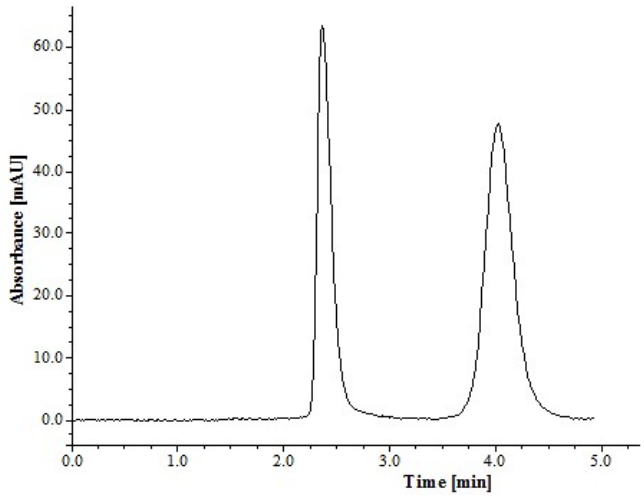

Fig. S4 Separation chromatogram of vitamin-C and aspirin extracted from Aspirin-C tablets using C_2 column. Conditions: mobile phase: acetonitrile/water with 0.1% formic acid (75/25, v/v), flow rate: 0.35 mL min^{-1}, detection wavelength: UV at 230 nm, column temperature: 30^{o}C. Peaks identification by order of elution: 1- vitamin-C and 2- aspirin.

Fig. S5

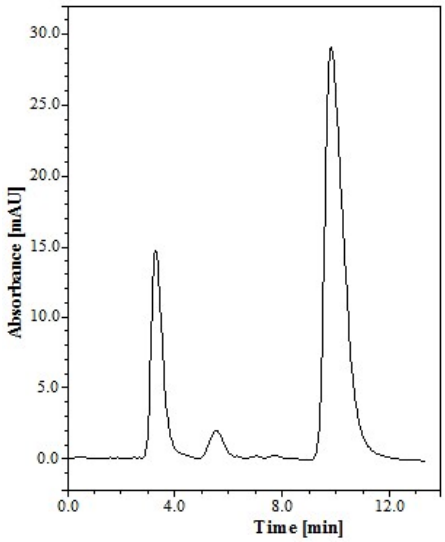

Fig. S5 Separation chromatogram of the black tea hot water extract using C_2 column. Conditions: mobile phase: acetonitrile/water with 0.1% formic acid (35/65, v/v), flow rate: 0.20 mL min^{-1}, detection wavelength: UV at 273 nm, column temperature: 30^{o}C. Peaks identification by order of elution: 1- gallic acid, 2- theobromine, and 3- caffeine.

Table S2

Table S2 Comparison of the prepared columns with other reported HPLC methods (published in 2014 and beyond) for the separation of PAH compounds.

Stationary phases	Analytes	Column (length × i.d. mm)	Mobile phase composition & mode	Flow rate (mL min^{-1})	Chromatographic evaluation[1]	Run time (min)	Ref.
Zorbax SB-C18, 3.5 µm	5 PAHs	100 × 2.1	Acetonitrile/water Isocratic elution	0.2	ND[2]	8	[1]
Eclipse XDB-C18, 5 µm	6 PAHs	150 × 4.6	Methanol/water Isocratic elution	0.5	ND	26	[2]
Acquity UPLC BEH C18, 1.7 µm	4 PAHs	50 × 2.1	Acetonitrile/water Isocratic elution	0.5	ND	4.5	[3]
Poly(benzyl methacrylate)	3 PAHs	200 × 0.32	Acetonitrile/water Isocratic elution	6 µL min^{-1}	R_s: 5.2–5.8 N: 5,520–10,180 plates m^{-1}	16	[4]
H5-ODS C18, 5 µm	5 PAHs	150 × 4.6	Acetonitrile/water Isocratic elution	0.8	ND	19	[5]
Sporopollenin-poly(hexyl methacrylate)	3 PAHs	150 × 0.32	Acetonitrile/water Isocratic elution	40 µL min^{-1}	R_s: 1.7–4.2 N: 1,072– 5,347 plates m^{-1}	7.5	[6]
Zorbax Eclipse Plus C18, 5 µm	5 PAHs	150 × 4.6	Methanol/water Isocratic elution	1.0	ND	8	[7]
Sporopollenin-poly(hexyl methacrylate)	3 PAHs	150 × 0.32	Acetonitrile/water Isocratic elution	15 µL min^{-1}	R_s: 1.52–1.74 N: 3,000–13,100 plates m^{-1}	1.75	[8]
Spheri-5 ODS, 5 µm	4 PAHs	250 × 4.6	Methanol/water Isocratic elution	1.0	R_s >1.0	32	[9]
Hypersil Green PAH, 5 µm	5 PAHs	250 × 4.6	Acetonitrile/water Gradient elution	1.0	ND	28	[10]
CNT-poly(benzyl methacrylate)	10 PAHs	200 × 0.1	Acetonitrile/water Gradient elution	0.5 µL min^{-1}	R_s: 1.74–3.98 N: 11,500–41,700 plates m^{-1}	13	[11]
LiChrospher 100 RP-8 endcapped, 5 µm	5 PAHs	150 × 4.6	Acetonitrile/water Isocratic elution	1.0	ND	10	[12]
HALO 90 Å PAH, 2.7 µm	16 PAHs	50 × 4.6	Acetonitrile/water Gradient elution	2.0	R_s: ≥1.13	5	[13]
ZORBAX Eclipse 95 Å PAH, 1.8 µm	16 PAHs	50 × 4.6	Acetonitrile/water Gradient elution	3.0	R_s: ≥1.36	3	[13]
Kinetex EVO-C18, 5 µm	10 PAHs	150 × 4.6	Acetonitrile/ methanol/water Isocratic elution	1.5	R_s: ≥1.5	16	[14]
Eclipse Plus C18, 5 µm	7 PAHs	150 × 4.6	Acetonitrile/water Gradient elution	1.0	ND	29	[15]
MIL-53(Al) MOF	7 PAHs	100 × 4.6	Acetonitrile/water Gradient elution	0.35	R_s: 2.02–11.05 N: 5700–63200 plates m^{-1}	22	[16]
Zorbax Eclipse PAH, 3.5 µm	24 PAHs	100 × 2.1	Acetonitrile/water Isocratic elution	0.237	ND	80	[17]
Poroshell 120 EC-C18, 2.7 µm	24 PAHs	50 × 2.1	Acetonitrile/water Isocratic elution	0.131	ND	25	[17]
Kinetex PAH, 3.5 µm	17 PAHs	150 × 4.6	Acetonitrile/water Gradient elution	1.0	ND	20	[18]
Montmorillonite-poly(glycidyl methacrylate)	7 PAHs	220 × 0.25	Acetonitrile/water Gradient elution	0.5 µL min^{-1}	R_s: 1.85–5.83 H: 17–42 µm	12	[19]
Supelcosil LC-PAH, 5 µm	15 PAHs	250 × 4.6	Acetonitrile/water Gradient elution	1.0	ND	22.5	[20]

Zorbax Eclipse C18 Plus, 5 µm	4 PAHs	250 × 4.6	Acetonitrile/water Gradient elution	1.0	ND	35	[21]
Agilent C18, 5 µm	24 PAHs	250 × 4.6	Acetonitrile/water Gradient elution	2.2	ND	26	[22]
Pursuit PAH, 5 µm	11 PAHs	250 × 4.6	Acetonitrile/water Gradient elution	0.5	ND	36	[23]
Zorbax Eclipse PAH, 1.8 µm	15 PAHs	50 × 4.6	Acetonitrile/water Gradient elution	1.0	ND	14	[24]
Nucleosil C18 PAH, 5 µm	8 PAHs	150 × 4.0	Acetonitrile/water Gradient elution	0.8	ND	45	[25]
Silica-UIO-66 composite	3 PAHs	100 × 2.1	Hexane Isocratic elution	0.4	R_s: ≥0.886	8	[26]
Column C_2[3] Column C_3[4]	9 PAHs	50 × 2.1	Acetonitrile/water Gradient elution	0.1	R_s: 1.11–4.63 H: 4.08–39.49 µm	32	this work

[1]Chromatographic evaluation: Main chromatographic parameters measured in the study (if any). [2]ND: Not determined. [3]Poly(itaconic anhydride-co-ethylene dimethacrylate) modified with octadecylamine. [4]Poly(itaconic anhydride-co-ethylene dimethacrylate) modified with benzylamine.

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