Supplementary data

Surfactant-enhanced air-agitation liquid-liquid microextraction of polycyclic aromatic hydrocarbons from edible oil samples using magnetic deep eutectic solvent prior to HPLC

Fariba Adib[1,2], Mohammad Reza Afshar Mogaddam[3, 4,*], Mahboob Nemati [1,3, *], Mir Ali Farajzadeh[5, 6], Ali Mohebbi[3], Ali Akbar Alizadeh Nabil[3]

[1]*Department of Food and Drug Control, Faculty of Pharmacy, Tabriz University of Medical Science, Tabriz, Iran*

[2] *Neurosciences Research Center, Tabriz University of Medical Sciences, Tabriz, Iran*

[3] *Food and Drug Safety Research Center, Tabriz University of Medical Science, Tabriz, Iran*

[4] *Pharmaceutical Analysis Research Center, Tabriz University of Medical Science, Tabriz, Iran*

[5] *Department of Analytical Chemistry, Faculty of Chemistry, University of Tabriz, Tabriz, Iran*

[6]*Engineering Faculty, Near East University, 99138 Nicosia, North Cyprus, Mersin 10, Turkey*

*Corresponding author: Prof. M. Nemati

Email addresses: nematim@tbzmed.ac.ir; mahnema@gmail.com

Corresponding author: Dr. M.R. Afshar Mogaddam

E–mail addresses: mr.afsharmogaddam@yahoo.com; Afsharmogaddam@tbzmed.ac.ir

Tel.: +98 4133372250

Fax: 98 4133344798

Table S1. Experimental factors, levels, and the obtained results for CCD design.

Variables	levels				
	-α	-1	0	+1	+α
(A) Vortexing time (min)	1	2	3	4	5
(B) MDES volume (µL)	65	72	78	83	90
(C) Extraction number	1	4	6	7	10
(D) SDS solution concentration (% w/v)	0	0.3	0.5	0.7	1.0
(E) SDS solution volume (µL)	0	87	150	213	300

Std	Run	A	B	C	D	E	Average ER (%) (n=3) obtained from three repeated determinations of				
							Acenaphtene	Phentaphtrene	Anthracene	Pyrene	Benzo (a) pyrene
6	1	4	72	7	0.3	87	34.6	30.4	29.6	27.4	28.8
28	2	4	83	4	0.7	213	24.5	28.2	27.5	25.4	26.7
14	3	4	72	7	0.7	87	51.3	59.0	57.5	53.2	55.9
4	4	4	83	4	0.3	87	53.1	61.1	59.5	55.1	57.9
37	5	3	78	1	0.5	150	28.0	32.2	31.4	29.1	30.6
1	6	2	72	4	0.3	87	28.0	32.2	31.4	29.1	30.6
46	7	3	78	6	0.5	150	35.5	40.8	39.8	36.8	38.7
11	8	2	83	4	0.7	87	23.3	34.7	33.8	31.3	32.9
32	9	4	83	7	0.7	213	26.3	28.0	27.2	25.2	26.5
38	10	3	78	10	0.5	150	29.1	33.5	32.6	30.2	31.7
41	11	3	78	6	0.5	0	53.4	61.4	59.8	55.4	58.2
29	12	2	72	7	0.7	213	53.6	64.6	62.9	58.3	61.3
25	13	2	72	4	0.7	213	39.0	39.3	38.3	35.5	37.3
3	14	2	83	4	0.3	87	43.9	50.5	49.2	45.5	47.9
33	15	1	78	6	0.5	150	33.2	38.2	37.2	34.4	36.2
35	16	3	65	6	0.5	150	45.3	52.1	50.7	47.0	49.4
8	17	4	83	7	0.3	87	40.3	46.3	45.1	41.8	43.9
27	18	2	83	4	0.7	213	39.4	50.8	49.4	45.8	48.1
18	19	4	72	4	0.3	213	34.9	40.1	39.1	36.2	38.0
17	20	2	72	4	0.3	213	42.3	48.6	47.4	43.9	46.1
43	21	3	78	6	0.5	150	36.5	41.9	40.8	37.8	39.8
22	22	4	72	7	0.3	213	39.7	48.6	47.4	43.9	46.1
50	23	3	78	6	0.5	150	32.6	28.2	27.5	25.4	26.7
23	*24*	*2*	*83*	*7*	*0.3*	*213*	*77.1*	*88.7*	*86.4*	*79.9*	*84.0*
39	25	3	78	6	0.0	150	52.0	59.8	58.2	53.9	56.7
42	26	3	78	6	0.5	300	74.1	85.2	83.0	76.8	80.8
9	27	2	72	4	0.7	87	30.9	28.2	27.5	25.4	26.7
10	28	4	72	4	0.7	87	56.4	64.8	63.1	58.5	61.5
45	29	3	78	6	0.5	150	33.7	38.8	37.8	35.0	36.7
12	30	4	83	4	0.7	87	26.4	30.4	29.6	27.4	28.8
34	31	5	78	6	0.5	150	36.5	41.9	40.8	37.8	39.8
24	32	4	83	7	0.3	213	53.7	68.1	66.3	61.4	64.5
30	33	4	72	7	0.7	213	44.6	51.3	50.0	46.3	48.6
7	34	2	83	7	0.3	87	44.0	50.6	49.3	45.6	48.0
26	35	4	72	4	0.7	213	36.5	42.0	40.9	37.8	39.8
13	36	2	72	7	0.7	87	30.8	35.4	34.5	31.9	33.6
16	37	4	83	7	0.7	87	23.5	27.0	26.3	24.4	25.6
2	38	4	72	4	0.3	87	47.3	54.4	53.0	49.0	51.5
48	39	3	78	6	0.5	150	31.2	35.9	34.9	32.4	34.0
31	40	2	83	7	0.7	213	48.2	55.4	54.0	50.0	52.5
47	41	3	78	6	0.5	150	35.0	40.3	39.2	36.3	38.1
5	42	2	72	7	0.3	87	20.6	19.8	19.3	17.8	18.7
40	43	3	78	6	1.0	150	28.2	32.4	31.6	29.2	30.7
20	44	4	83	4	0.3	213	52.4	60.3	58.7	54.3	57.1
36	45	3	90	6	0.5	150	42.3	48.6	47.4	43.9	46.1
49	46	3	78	6	0.5	150	32.6	43.5	42.3	39.2	41.2
21	47	2	72	7	0.3	213	52.8	68.1	66.3	61.4	64.5
19	48	2	83	4	0.3	213	70.1	80.6	78.5	72.7	76.4
44	49	3	78	6	0.5	150	43.0	30.7	29.9	27.7	29.1
15	50	2	83	7	0.7	87	20.8	23.9	23.3	21.6	22.7

Table S2. Results of ANOVA for CCD design for acenaphtene.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	8067.09	20	403.35	55.97	< 0.0001	Significant
A-Vortexing time	3.08	1	3.08	0.4275	0.5184	
B-Solvent volume	6.34	1	6.34	0.8791	0.3562	
C-Extraction number	5.99	1	5.99	0.8309	0.3695	
D-Surfactant percent	1076.04	1	1076.04	149.30	< 0.0001	
E-Surfactant volume	1010.82	1	1010.82	140.26	< 0.0001	
AB	404.89	1	404.89	56.18	< 0.0001	
AC	73.68	1	73.68	10.22	0.0033	
AD	21.95	1	21.95	3.05	0.0915	
AE	1254.00	1	1254.00	174.00	< 0.0001	
BC	4.55	1	4.55	0.6307	0.4336	
BD	1874.86	1	1874.86	260.14	< 0.0001	
BE	166.23	1	166.23	23.07	< 0.0001	
CD	32.06	1	32.06	4.45	0.0437	
CE	315.35	1	315.35	43.76	< 0.0001	
DE	121.87	1	121.87	16.91	0.0003	
A^2	0.3539	1	0.3539	0.0491	0.8262	
B^2	125.87	1	125.87	17.47	0.0002	
C^2	78.31	1	78.31	10.87	0.0026	
D^2	40.24	1	40.24	5.58	0.0251	
E^2	1406.87	1	1406.87	195.21	< 0.0001	
Residual	209.00	29	7.21			
Lack of Fit	115.77	22	5.26	0.3951	0.9548	Not significant
Pure Error	93.24	7	13.32			
Cor Total	8276.09	49				

Table S3. Results of ANOVA for CCD design for phentaphtrene.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	11626.99	20	581.35	19.98	< 0.0001	Significant
A-Vortexing time	11.89	1	11.89	0.4089	0.5276	
B-Solvent volume	56.15	1	56.15	1.93	0.1753	
C-Extraction number	11.02	1	11.02	0.3790	0.5430	
D-Surfactant percent	1446.49	1	1446.49	49.73	< 0.0001	
E-Surfactant volume	1691.05	1	1691.05	58.13	< 0.0001	
AB	614.72	1	614.72	21.13	< 0.0001	
AC	128.05	1	128.05	4.40	0.0447	
AD	24.72	1	24.72	0.8498	0.3642	
AE	1617.49	1	1617.49	55.60	< 0.0001	
BC	40.42	1	40.42	1.39	0.2481	
BD	2280.03	1	2280.03	78.38	< 0.0001	
BE	101.38	1	101.38	3.49	0.0721	
CD	35.20	1	35.20	1.21	0.2804	
CE	673.13	1	673.13	23.14	< 0.0001	
DE	323.61	1	323.61	11.12	0.0023	
A^2	11.15	1	11.15	0.3832	0.5407	
B^2	286.46	1	286.46	9.85	0.0039	
C^2	37.87	1	37.87	1.30	0.2632	
D^2	128.10	1	128.10	4.40	0.0447	
E^2	2223.84	1	2223.84	76.45	< 0.0001	
Residual	843.60	29	29.09			
Lack of Fit	632.88	22	28.77	0.9557	0.5713	Not significant
Pure Error	210.71	7	30.10			
Cor Total	12470.59	49				

Table S4. Results of ANOVA for CCD design for anthracene.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	11028.28	20	551.41	19.98	< 0.0001	significant
A-Vortexing time	11.28	1	11.28	0.4089	0.5276	
B-MDES volume	53.26	1	53.26	1.93	0.1753	
C-Extraction number	10.46	1	10.46	0.3790	0.5430	
D-SDS solution concentration	1372.01	1	1372.01	49.73	< 0.0001	
E-SDS solution volume	1603.98	1	1603.98	58.13	< 0.0001	
AB	583.06	1	583.06	21.13	< 0.0001	
AC	121.46	1	121.46	4.40	0.0447	
AD	23.45	1	23.45	0.8498	0.3642	
AE	1534.20	1	1534.20	55.60	< 0.0001	
BC	38.34	1	38.34	1.39	0.2481	
BD	2162.63	1	2162.63	78.38	< 0.0001	
BE	96.16	1	96.16	3.49	0.0721	
CD	33.39	1	33.39	1.21	0.2804	
CE	638.47	1	638.47	23.14	< 0.0001	
DE	306.95	1	306.95	11.12	0.0023	
A^2	10.57	1	10.57	0.3832	0.5407	
B^2	271.71	1	271.71	9.85	0.0039	
C^2	35.92	1	35.92	1.30	0.2632	
D^2	121.50	1	121.50	4.40	0.0447	
E^2	2109.32	1	2109.32	76.45	< 0.0001	
Residual	800.16	29	27.59			
Lack of Fit	600.29	22	27.29	0.9557	0.5713	not significant
Pure Error	199.86	7	28.55			
Cor Total	11828.43	49				

Table S5. Results of ANOVA for CCD design for pyrene.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	9452.47	20	472.62	19.98	< 0.0001	significant
A-Vortexing time	9.67	1	9.67	0.4089	0.5276	
B-MDES volume	45.65	1	45.65	1.93	0.1753	
C-Extraction number	8.96	1	8.96	0.3790	0.5430	
D-SDS solution concentration	1175.97	1	1175.97	49.73	< 0.0001	
E-SDS solution volume	1374.79	1	1374.79	58.13	< 0.0001	
AB	499.75	1	499.75	21.13	< 0.0001	
AC	104.10	1	104.10	4.40	0.0447	
AD	20.10	1	20.10	0.8498	0.3642	
AE	1314.98	1	1314.98	55.60	< 0.0001	
BC	32.86	1	32.86	1.39	0.2481	
BD	1853.61	1	1853.61	78.38	< 0.0001	
BE	82.42	1	82.42	3.49	0.0721	
CD	28.62	1	28.62	1.21	0.2804	
CE	547.24	1	547.24	23.14	< 0.0001	
DE	263.09	1	263.09	11.12	0.0023	
A^2	9.06	1	9.06	0.3832	0.5407	
B^2	232.88	1	232.88	9.85	0.0039	
C^2	30.78	1	30.78	1.30	0.2632	
D^2	104.14	1	104.14	4.40	0.0447	
E^2	1807.93	1	1807.93	76.45	< 0.0001	
Residual	685.82	29	23.65			
Lack of Fit	514.52	22	23.39	0.9557	0.5713	not significant
Pure Error	171.30	7	24.47			
Cor Total	10138.29	49				

Table S6. Results of ANOVA for CCD design for benzo (a) pyrene.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	10445.39	20	522.27	19.98	< 0.0001	Significant
A-Vortexing time	10.68	1	10.68	0.4089	0.5276	
B-MDES volume	50.44	1	50.44	1.93	0.1753	
C-Extraction number	9.90	1	9.90	0.3790	0.5430	
D-SDS solution concentration	1299.49	1	1299.49	49.73	< 0.0001	
E-SDS solution volume	1519.20	1	1519.20	58.13	< 0.0001	
AB	552.25	1	552.25	21.13	< 0.0001	
AC	115.04	1	115.04	4.40	0.0447	
AD	22.21	1	22.21	0.8498	0.3642	
AE	1453.11	1	1453.11	55.60	< 0.0001	
BC	36.31	1	36.31	1.39	0.2481	
BD	2048.32	1	2048.32	78.38	< 0.0001	
BE	91.07	1	91.07	3.49	0.0721	
CD	31.62	1	31.62	1.21	0.2804	
CE	604.72	1	604.72	23.14	< 0.0001	
DE	290.73	1	290.73	11.12	0.0023	
A^2	10.02	1	10.02	0.3832	0.5407	
B^2	257.35	1	257.35	9.85	0.0039	
C^2	34.02	1	34.02	1.30	0.2632	
D^2	115.08	1	115.08	4.40	0.0447	
E^2	1997.84	1	1997.84	76.45	< 0.0001	
Residual	757.86	29	26.13			
Lack of Fit	568.56	22	25.84	0.9557	0.5713	Not significant
Pure Error	189.30	7	27.04			
Cor Total	11203.25	49				