

Supplementary Material

Fast preconcentration of Pb(II) and Cu(II) in liquid milk using syringe solid-phase extraction on alginate and PVA biopolymer loaded with activated carbon.

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The data for the appendix section

Table

Table S1. Elemental composition in the material from EDX results

Material	Atom (%)					
	C	O	N	Ca	Pb	Cu
ACP	44.6	40.6	11.7	2.89	-	-
ACP after Pb adsorption	52.9	34.0	10.8	1.43	0.81	-
ACP after Pb desorption	47.5	41.8	10.7	0.03	0.01	-
ACP after Cu adsorption	45.4	40.4	11.9	1.91	-	0.46
ACP after Cu desorption	48.5	40.6	10.2	0.63	-	0.09

Table S2. Results of surface area and pore volume for each material from SAA BET test.

Material	Sbet m ² /g	Vtotal cm ³ /g	Vmicro cm ³ /g	Micropore %	Mesopore %
Carbon	587	0.52	0.17	31.9	68.1
ACP	198	0.19	0.06	30.1	69.9
ACP after Pb adsorption	197	0.19	0.05	29.0	71.0
ACP after Pb desorption 10x	454	0.42	0.17	39.7	60.3
ACP after Cu adsorption	187	0.13	0.04	31.9	68.1
ACP after Cu desorption 10x	574	0.49	0.16	33.1	66.9

S BET: Specific surface area based on Brunauer–Emmett–Teller (BET) theory calculation.

V total: Total pore volume obtained from N₂ sorption isotherm data at relative pressure of 0.99. V micro: Micropore volume calculated from t-plot.

Table S3. The design of optimization for Pb adsorption

Std Run	Factor 1 A:pH	Factor 2 B:Mass ACP mg	Factor 3 C:Cycle of sample Time	Response 1 Adsorption %	
1	10	3	25	4	77.7
2	17	7	25	4	60.3
3	2	3	125	4	87.5
4	4	7	125	4	73.7
5	1	3	75	2	84.3
6	12	7	75	2	70.7
7	9	3	75	6	88.8
8	3	7	75	6	73.2
9	11	5	25	2	84.3
10	5	5	125	2	95.8
11	16	5	25	6	87.8
12	7	5	125	6	97.9
13	14	5	75	4	99.8
14	13	5	75	4	98.8
15	6	5	75	4	99.3

16	8	5	75	4	99.5
17	15	5	75	4	99.3

Table S4. The design of optimization for Pb desorption

		Factor 1	Factor 2	Factor 3	Response 1
Std	Run	A:Cycle of eluent times	B:Concentration HNO3 mol/L	C:Factor preconcentration times	Desorption %
1	8	7	0.5	62.5	75.2
2	16	11	0.5	62.5	86.5
3	10	7	2	62.5	84.7
4	5	11	2	62.5	93.4
5	14	7	1.25	25	86.1
6	4	11	1.25	25	93.6
7	2	7	1.25	100	87.5
8	1	11	1.25	100	98.6
9	7	9	0.5	25	86.3
10	6	9	2	25	94.3
11	13	9	0.5	100	89.1
12	9	9	2	100	98.2
13	3	9	1.25	62.5	97.7
14	12	9	1.25	62.5	97.7
15	11	9	1.25	62.5	97.7
16	15	9	1.25	62.5	96.9
17	17	9	1.25	62.5	96.8

Table S5. The design of optimization for Cu adsorption

		Factor 1	Factor 2	Factor 3	Response 1
Std	Run	A:pH	B:Mass ACP mg	C:Cycle sample times	Adsorption %
1	12	2	25	4	10.3
2	15	6	25	4	79.6
3	11	2	125	4	18.1
4	1	6	125	4	91.8
5	10	2	75	2	13.6
6	14	6	75	2	85.3
7	7	2	75	6	19.3
8	4	6	75	6	90.1
9	3	4	25	2	82.6
10	5	4	125	2	90.3
11	6	4	25	6	86.4
12	16	4	125	6	98.7
13	8	4	75	4	99.1
14	17	4	75	4	99.4
15	2	4	75	4	99
16	13	4	75	4	99.4
17	9	4	75	4	99.1

Table S6. The design of optimization for Cu desorption

		Factor 1	Factor 2	Factor 3	Response 1
Std	Run	A:Cycle of eluent Times	B:Concentration HNO3 Mol/ L	C:Factor preconcentration Times	desorption %
1	13	6	0.5	62.5	73

2	8	10	0.5	62.5	80.2
3	2	6	2	62.5	91.4
4	15	10	2	62.5	97.2
5	4	6	1.25	25	88.2
6	1	10	1.25	25	94.4
7	17	6	1.25	100	91.2
8	5	10	1.25	100	99.1
9	10	8	0.5	25	77.3
10	14	8	2	25	93.4
11	16	8	0.5	100	80.6
12	9	8	2	100	98.9
13	12	8	1.25	62.5	96.7
14	11	8	1.25	62.5	96.4
15	3	8	1.25	62.5	96.4
16	6	8	1.25	62.5	96.1
17	7	8	1.25	62.5	96.7

Table S7. ANOVA for Quadratic model of Pb adsorption

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	2407.67	9	267.52	1172.59	< 0.0001 significant
A-pH	456.02	1	456.02	1998.84	< 0.0001
B-Mass ACP	250.88	1	250.88	1099.66	< 0.0001
C-Cycle of sample	19.84	1	19.84	86.98	< 0.0001
AB	3.24	1	3.24	14.20	0.0070
AC	1.0000	1	1.0000	4.38	0.0746
BC	0.4900	1	0.4900	2.15	0.1862
A ²	1420.87	1	1420.87	6227.99	< 0.0001
B ²	160.29	1	160.29	702.59	< 0.0001
C ²	12.46	1	12.46	54.60	0.0002
Residual	1.60	7	0.2281		
Lack of Fit	1.06	3	0.3550	2.67	0.1833 not significant
Pure Error	0.5320	4	0.1330		
Cor Total	2409.26	16			

Fit Statistics

Std. Dev.	0.4776	R²	0.9993
Mean	86.98	Adjusted R²	0.9985
C.V. %	0.5491	Predicted R²	0.9926
		Adeq Precision	105.3406

Table S8. ANOVA for Quadratic model of Pb desorption

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	696.08	9	77.34	455.14	< 0.0001 significant
A-Cycle of eluent	186.24	1	186.24	1096.02	< 0.0001
B-Concentration HNO ₃	140.28	1	140.28	825.53	< 0.0001
C-Factor preconcentration	21.45	1	21.45	126.24	< 0.0001
AB	1.69	1	1.69	9.95	0.0161
AC	3.24	1	3.24	19.07	0.0033
BC	0.3025	1	0.3025	1.78	0.2239

A ²	176.12	1	176.12	1036.44	< 0.0001
B ²	148.69	1	148.69	875.00	< 0.0001
C ²	1.31	1	1.31	7.70	0.0275
Residual	1.19	7	0.1699		
Lack of Fit	0.3175	3	0.1058	0.4855	0.7105 not significant
Pure Error	0.8720	4	0.2180		
Cor Total	697.26	16			

Fit Statistics

Std. Dev.	0.4122	R²	0.9983
Mean	91.78	Adjusted R²	0.9961
C.V. %	0.4491	Predicted R²	0.9908
		Adeq Precision	74.4082

Table S9. ANOVA for Quadratic model of Cu adsorption

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	18835.17	9	2092.80	27510.94	< 0.0001 significant
A-pH	10188.78	1	10188.78	1.339E+05	< 0.0001
B-Mass ACP	200.00	1	200.00	2629.11	< 0.0001
C-Cycle sample	64.41	1	64.41	846.72	< 0.0001
AB	4.84	1	4.84	63.62	< 0.0001
AC	0.2025	1	0.2025	2.66	0.1468
BC	5.29	1	5.29	69.54	< 0.0001
A ²	7907.95	1	7907.95	1.040E+05	< 0.0001
B ²	147.19	1	147.19	1934.89	< 0.0001
C ²	60.40	1	60.40	794.00	< 0.0001
Residual	0.5325	7	0.0761		
Lack of Fit	0.3925	3	0.1308	3.74	0.1176 not significant
Pure Error	0.1400	4	0.0350		
Cor Total	18835.70	16			

Fit Statistics

Std. Dev.	0.2758	R²	1.0000
Mean	74.24	Adjusted R²	0.9999
C.V. %	0.3715	Predicted R²	0.9997
		Adeq Precision	419.9620

Table S8. ANOVA for Quadratic model of Cu desorption

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	1076.94	9	119.66	1232.70	< 0.0001 significant
A-Cycle of eluent	91.80	1	91.80	945.71	< 0.0001
B-Concentration HNO ₃	609.01	1	609.01	6273.78	< 0.0001
C-Factor preconcentration	34.03	1	34.03	350.58	< 0.0001
AB	0.4900	1	0.4900	5.05	0.0595
AC	0.7225	1	0.7225	7.44	0.0294
BC	1.21	1	1.21	12.47	0.0096
A ²	29.96	1	29.96	308.64	< 0.0001
B ²	293.04	1	293.04	3018.82	< 0.0001

C ²	1.36	1	1.36	13.97	0.0073
Residual	0.6795	7	0.0971		
Lack of Fit	0.4275	3	0.1425	2.26	0.2234 not significant
Pure Error	0.2520	4	0.0630		
Cor Total	1077.62	16			

Fit Statistics

Std. Dev.	0.3116	R²	0.9994
Mean	91.01	Adjusted R²	0.9986
C.V. %	0.3423	Predicted R²	0.9933
	Adeq Precision		109.2766

Figure

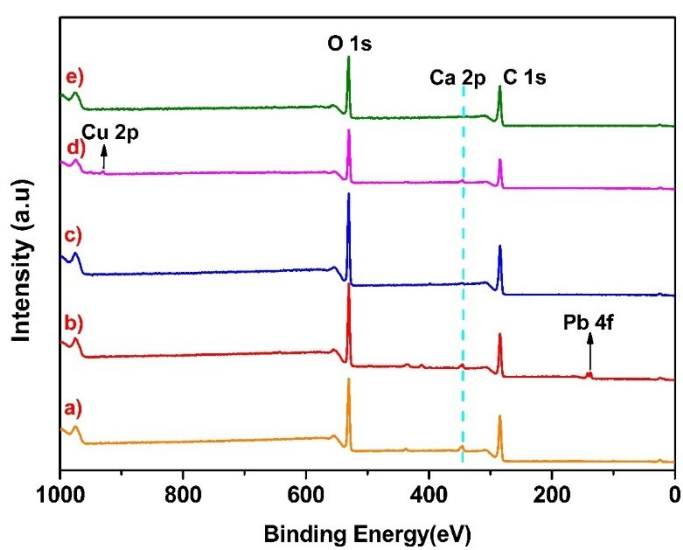


Figure S1. The XPS survey from ACP before and after applications