Supplementary Information (SI) for Analytical Methods.
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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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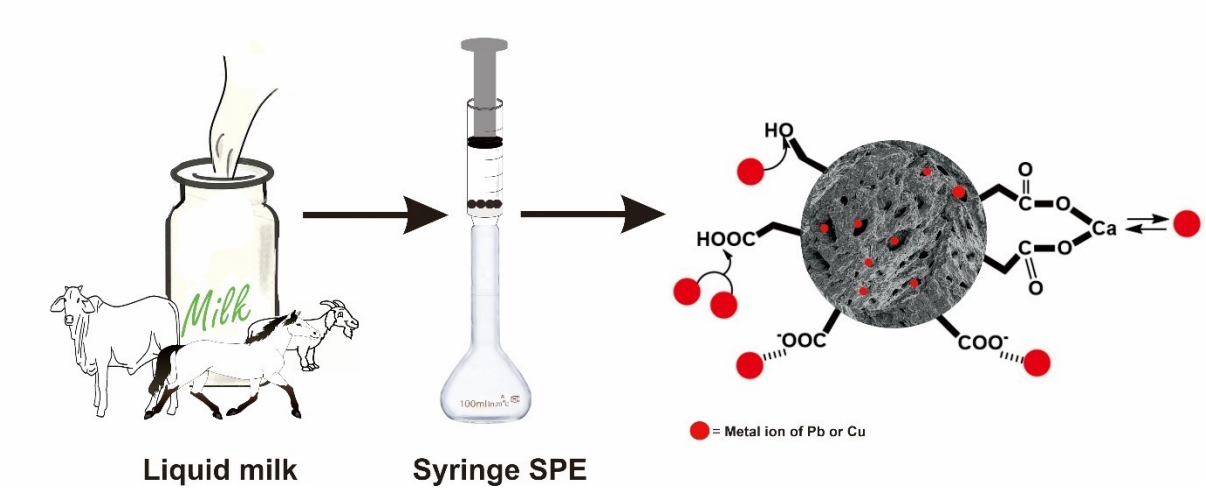
Grapical abstract



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	Vtotal	Vmicro	Micropore	Mesopore
	m^2/g	cm^3/g	cm^3/g	%	%
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

Std	Run	Factor 1 A:Cycle of eluent times	Factor 2 B:Concentration HNO3 mol/L	Factor 3 C:Factor preconcentration times	Response 1 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

Std	Run	Factor 1 A:pH	Factor 2 B:Mass ACP mg	Factor 3 C:Cycle sample times	Response 1 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

Std	Run	Factor 1 A:Cycle of eluent Times	Factor 2 B:Concentartion HNO3 Mol/ L	Factor 3 C:Factor preconcentration Times	Response 1 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	**R^2**	0.9993
Mean	86.98	**Adjusted R^2**	0.9985
C.V. %	0.5491	**Predicted R^2**	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 HNO3	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	**R^2**	0.9983
Mean	91.78	**Adjusted R^2**	0.9961
C.V. %	0.4491	**Predicted R^2**	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	**R^2**	1.0000
Mean	74.24	**Adjusted R^2**	0.9999
C.V. %	0.3715	**Predicted R^2**	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-Concentartion HNO3	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	**R^2**	0.9994
Mean	91.01	**Adjusted R^2**	0.9986
C.V. %	0.3423	**Predicted R^2**	0.9933
		Adeq Precision	109.2766

Figure

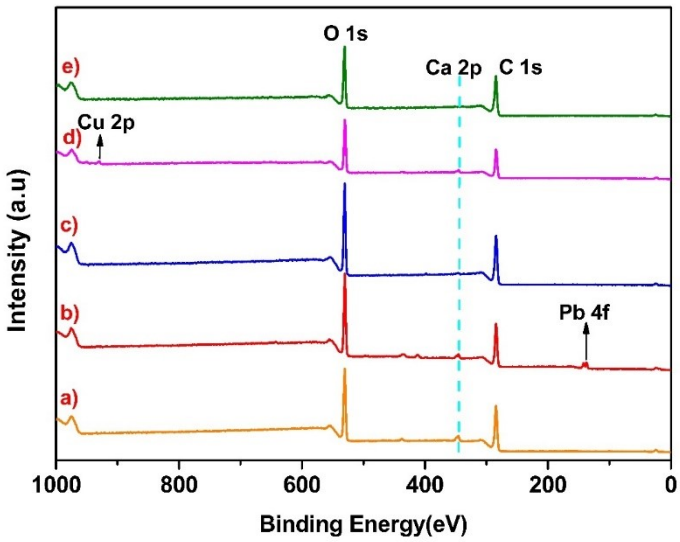


Figure S1. The XPS survey from ACP before and after applications