

Electronic Supplementary Information

Synthesis of new hybrid sorbent 2-mercaptobenzaldehyde SBA-15 and its application in solid phase extraction of Cd(II) from water and food samples

B. Sreenu^a, K. Imran^a, K. Seshaiyah^{*a}, Priti Sharma^b, and A. P. Singh^b

^aInorganic and Analytical Division, Department of Chemistry, Sri Venkateswara University, Tirupati, A.P-517502, India.

^bCatalysis Division, National Chemical Laboratory, Pune.

* Corresponding authors. Tel: +91877- 2289303. Email :seshaiyahsvu@gmail.com

S1 Determination of Cd(II) in water and vegetable samples

S 1.1 Collection and Preparation of water samples

The natural water samples were collected from two different places in Tirupati (A.P, India) in polyethylene bottles. The water samples were filtered through a 0.45µm cellulose membrane filter, spiked with known concentrations of Cd(II) and acidified to a pH of about 2.0 with concentrated HNO₃ and kept aside for overnight. 500ml of stored water sample was taken and pH was adjusted to 6.0 with 1:1 HCl or 1:1 ammonia followed by 2ml of acetate buffer and followed the procedure described in section 2.6 for determination of Cd concentration in each sample.

S 1.2 Collection of food samples

The vegetable samples namely, onion (*Alium cepa*), Lady's Finger (*Abelmoschus esculentus*), cucumber (*Cucumis sativus*) and tomato (*Solanum lycopersicum*) were collected in polyethylene bags from the nearby agricultural fields of Tirupati.

S 1.3 Sample preparation

The following procedure was used for food sample preparation. First, the collected vegetable samples were air dried for 5 days, then cleaned with double-distilled water followed by drying at 100 °C in a hot air oven for 3 h and then powdered. In second step, one gram of each sample was taken in PTFE digestion vessel, spiked with known concentrations

of Cd(II), homogenised and allowed for 24 h , and 5mL of concentrated nitric acid was added to each flask. After allowing for 10 h, the samples were digested in a microwave system for 10 min at 105 °C. A clear solution was obtained. The digested samples were further made up to 25 mL using double-distilled water and determined as described in section 2.5.

Table S1.a) Effect of the flow rates in the Cd(II) recovery.

Flow rate (mL/min)	% Recovery of Cd (II) ^a
0.5	94.3±2.5
1.5	95.2±4.8
2.0	94.8±3.1
2.5	95.7±3.5
3.0	95.1±4.2
3.5	94.8±3.4
4.0	94.2±3.8

b) Effect of eluent flow rate on recovery of Cd(II)

Flow rate (mL/min)	% Recovery of Cd (II) ^a
0.2	94.1±3.4
0.4	95.4±2.6
0.6	94.6±2.8
0.8	92.1±3.8
1.0	91.5±3.1

^a Average of four determinations±standard deviation.

Table S2. Effect of interfering ions on determination of Cd(II)

Foreign ions	Tolerance limit $\mu\text{g L}^{-1}$	Recovery (%)
Li^+	1600	94.2 \pm 1.0
Na^+	2100	93.8 \pm 1.0
K^+	1600	93.6 \pm 2.0
Mg^{+2}	1100	93.7 \pm 0.9
Ca^{+2}	1100	93.4 \pm 1.0
Al^{+3}	1800	94.0 \pm 2.0
Fe^{+3}	600	92.9 \pm 1.0
Zn^{+2}	450	92.5 \pm 0.8
Cr^{+3}	600	92.6 \pm 0.7
Cu^{+2}	350	93.0 \pm 0.6
Ni^{+2}	350	93.0 \pm 1.0
Mn^{2+}	150	92.6 \pm 0.7
CH_3COO^-	100	93.1 \pm 2.0
HCOO^-	100	93.6 \pm 1.0
Cl^-	600	94.0 \pm 1.0
SO_4^{-2}	500	93.5 \pm 0.5
NO_3^-	400	94.2 \pm 0.7
CO_3^{-2}	500	94.0 \pm 0.8
PO_4^{-2}	200	93.5 \pm 1.0

Table S3. Percentage of recovery of Cd(II) ions from the functionalized SBA-15 using different eluents.

Eluent Solution	Volume (mL)	% Recovery of Cd(II) (RSD)
CH ₃ COOH(M)	5	62.2 (0.8)
CH ₃ COOH(M)	15	66.5 (0.5)
CH ₃ COOH(M)	25	73.9 (1.2)
HCl(M)	5	62.8 (1.0)
HCl(M)	15	69.5 (0.6)
HCl(M)	25	75.9 (0.9)
HNO ₃ (M)	5	95.2 (1.0)
HNO ₃ (M)	15	95.6 (0.8)
HNO ₃ (M)	25	95.8 (1.3)

Table S4. Comparison of determination of Cd(II) by AAS method and ICPAES method

Vegetables samples(A. cepa (onion))			
S. No.	Spiked	AAS method($\mu\text{g/kg}$)	ICPAES method ($\mu\text{g/kg}$)
1	0	0.021
2	5	5.01 \pm 0.6	5.025 \pm 0.9
3	10	9.73 \pm 0.2	9.25 \pm 0.7
4	15	14.58 \pm 0.1	14.78 \pm 0.5

Table S5. Determination of Cd(II) in water and vegetable samples.

Sample	Spiked ($\mu\text{g/L}$)	Found ($\mu\text{g/L}$)	Recovery (%)
Water Sample.1	0.0	0.95 \pm 0.4	-
	5.0	5.56 \pm 0.2	93.44
	10.0	10.20 \pm 0.3	93.15
	15.0	15.09 \pm 0.1	94.60
Water Sample.2	0.0	0.92 \pm 0.2	-
	5.0	5.55 \pm 0.4	93.75
	10.0	10.10 \pm 0.3	92.49
	15.0	15.04 \pm 0.6	93.47
Vegetables	Spiked ($\mu\text{g/Kg}$)	Found ($\mu\text{g/Kg}$)	Recovery (%)
A. cepa (onion)	0.0	0.87 \pm 0.3	-
	5.0	5.45 \pm 0.2	92.84
	10.0	10.2 \pm 0.1	93.83
	15.0	15.01 \pm 0.4	94.58
A. esculentus (Lady's Finger)	0.0	0.89 \pm 0.2	-
	5.0	5.42 \pm 0.1	92.02
	10.0	10.15 \pm 0.3	93.20
	15.0	15.01 \pm 0.2	94.46
C. sativus (cucumber)	0.0	0.96 \pm 0.6	-
	5.0	5.57 \pm 0.3	93.29
	10.0	10.16 \pm 0.1	92.70
	15.0	15.02 \pm 0.1	94.11
L. esculentum (tomato)	0.0	0.94 \pm 0.2	-
	5.0	5.49 \pm 0.3	92.73
	10.0	10.18 \pm 0.1	93.31
	15.0	15.02 \pm 0.3	94.22