Electronic Supplementary Material (ESI) for New Journal of Chemistry.
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Trace amount determination of Cd(II), Pb(II) and Ni(II) ions in agricultural and seafood samples after magnetic solid phase extraction by MIL-101(Cr)/phenylthiosemicarbazide functionalized magnetite nanoparticles composite

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Real samples pretreatment

The fish, shrimp, and agricultural samples were purchased from local markets (Tehran, Iran). In the first step, each sample was washed with distilled water and then dried at 70 °C for 48 h. After chopping the dried samples (spiked or non-spiked), microwave-assisted acid digestion was conducted by addition of 2 mL distilled water, 4 mL HNO_3 65% w/w, and 2 mL H_2O_2 33% (w/v) to 0.5 g of the samples. Afterwards, the reactors containing the samples were exposed to the microwave radiation program (2 min at 250 W, no radiation for 2 min, 6 min at 250 W, 5 min at 400 W, 8 min at 550 W, and then venting for 8 min) [1]. Thereafter, the residue was diluted up to 100 mL using double distilled water. The certified reference material (CRM) was digested according to the outlined procedure.

[1] A.B. Yilmaz, *Environ. Res.* **2003**, *92*, 277.

Table 1S

The recoveries of target ions in the presence of potentially interfering ions.

Potentially interfering ions	Tolerable Concentration Ratio X[c]/ Cd, Pb, Ni	R[a] (%) ± S[b]		
		Cd	Ni	Pb
K^+	10000	100 ± 3	101 ± 4	99 ± 4
Na^+	10000	99 ± 4	100 ± 3	99 ± 2
Ca^{2+}	1000	98 ± 3	99 ± 3	96 ± 5
Al^{3+}	1000	97 ± 5	98 ± 4	95 ± 5
Co^{2+}	700	96 ± 5	95 ± 4	98 ± 5
Fe^{3+}	500	98 ± 3	99 ± 3	94 ± 5
Sn^{2+}	1000	99 ± 3	97 ± 3	99 ± 6
Mg^{2+}	1000	97 ± 5	98 ± 5	98 ± 5
Cu^{2+}	300	95 ± 4	95 ± 3	96 ± 5
Zn^{2+}	150	98 ± 5	97 ± 4	97 ± 3
Hg^{2+}	100	96 ± 4	98 ± 4	95 ± 6
Cr^{3+}	400	97 ± 6	95 ± 3	97 ± 4

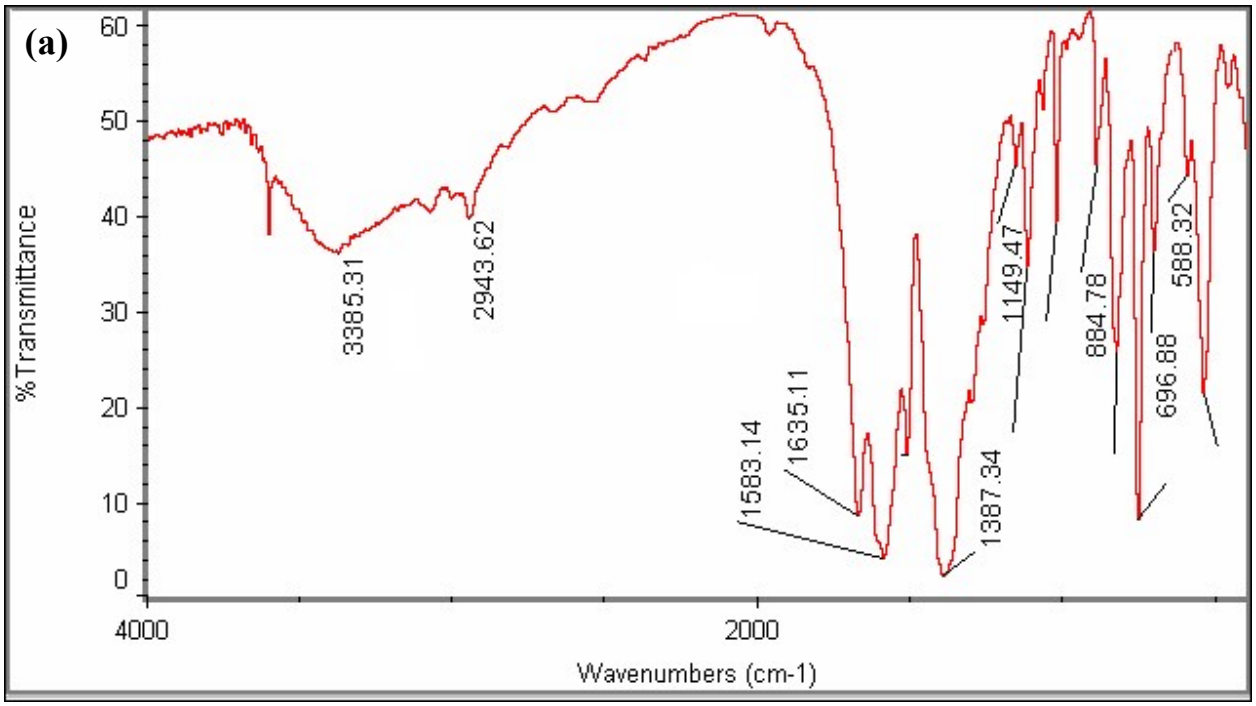
[a] Recovery

[b] standard deviation (n = 3)

Conditions: sample pH = 6.6, sample volume = 100 mL, sorbent amount = 13 mg, 100 μg L^{-1} of target ions, sorption time = 11.0 min, eluent = 2.2 mL, 0.85 mol/L HCl solution, elution time = 9.0 min.

[c] Concentration of potentially interfering ions.

Fig. 1S: FT-IR spectrum of the (a) prepared nanocomposite and (b) metals loaded nanocomposite.



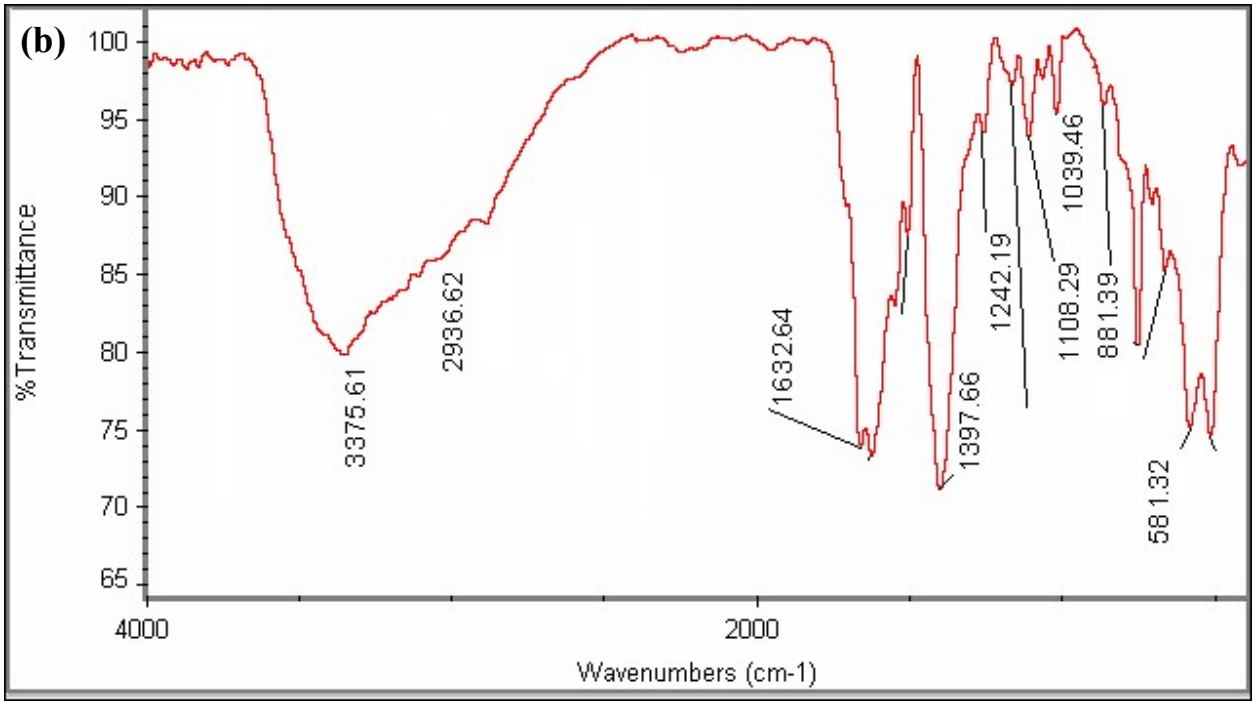


Fig. 2S: XRD pattern of (a) MIL-101(Cr) and MIL-101(Cr)/Fe_3O_4@SiO_2@PTSC composite.

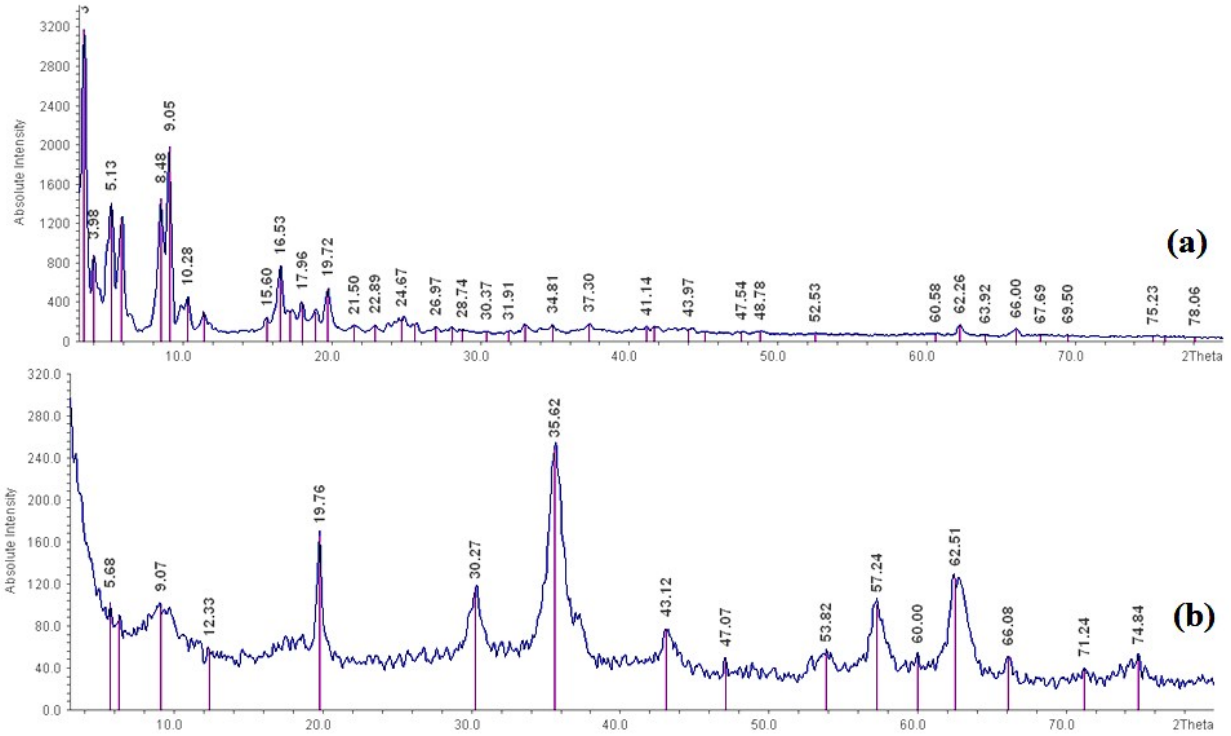
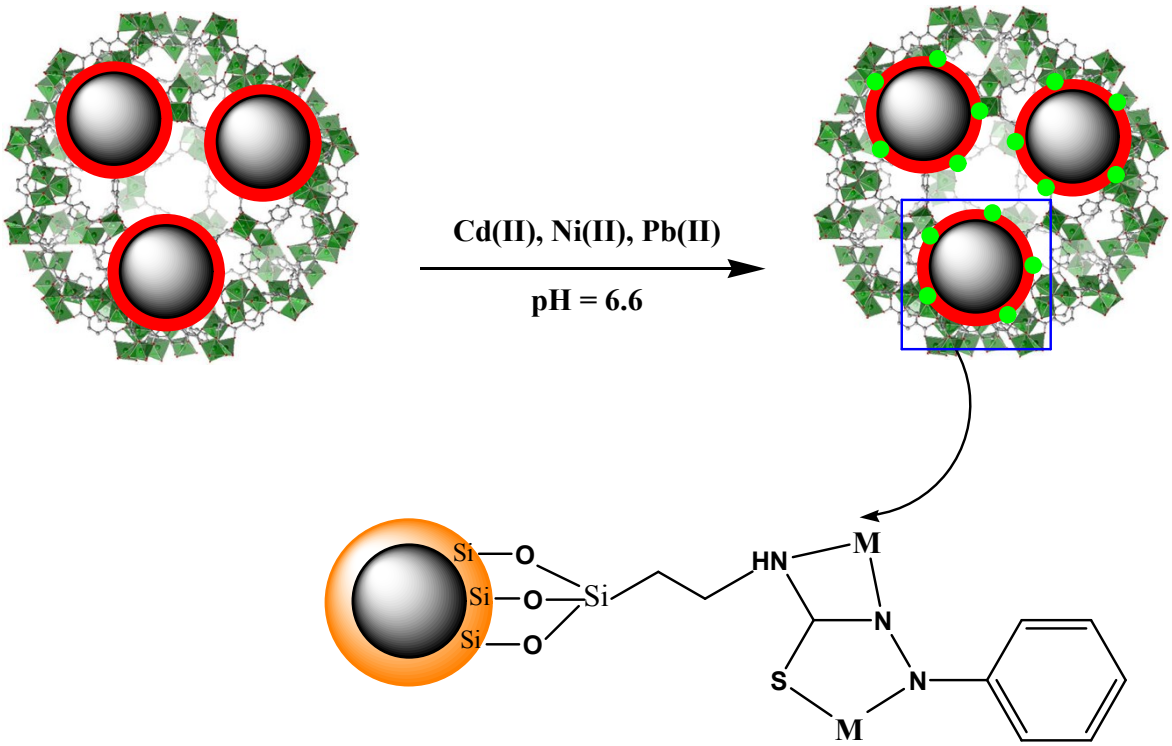


Fig. 3S: The proposed mechanism for the sorption of target analytes.



● **and M: Ni(II), Pb(II), Cd(II)**