3.1.6. FAAS evaluation

Flame atomic absorption spectrometry (FAAS) was used to assess the atomic composition of Fe_3O_4@Ni-Al-CO_3-LDH. For this purpose, 30 mg of the nanosorbent was dissolved with a few drops of concentrated HNO_3, and diluted to 50 mL with deionized water. The Ni and Al analyses were then performed by FAAS after appropriate dilutions with deionized water. According to the results obtained, the Ni^{2+}:Al^{3+} ratio (1.93:1) was in agreement with the expectations considering the proportion of metal salt precursors used in the LDH synthesis (2:1).

According to the results obtained, the adsorption of Cd(II) and Pb(II) ions onto the adsorbent may be attributed as follows: (i) contact between the target metal cations and the charge-compensating carbonate ion attached to the surface and edge and formation of the $CdCO_3$ and $PbCO_3$ precipitates (ii) substitution of the Ni(II) ions of the layers in the LDH structure with the Cd(II) and Pb(II) ions (iii) formation of the outer-sphere surface complexe Sur-O. . .Cd/Pb between the metal ions and some deprotonated hydroxyl groups (Sur–O−) (iv) surface adsorption derived from the nature of cations (e.g. electronegativity). The results obtained show that the surface-induced precipitation is a major mechanism for the metal-cation adsorption by LDH, which occurs due to localized high pH values and the released carbonate ions available to the metal cations.

Table 1S. Analytical characteristics of proposed method for Cd^{2+} and Pb^{2+} ions in optimum conditions.

Parameter	Cd^{2+}	Pb^{2+}
PF[a]	80	80
LDR[b]	0.75-35	4-370
LOD[c]	0.25	1.0
Intra-day precision (RSD[d] %)	3.4 (10 ng mL^{-1}) 3.2 (20 ng mL^{-1})	3.1 (50 ng mL^{-1}) 2.9 (100 ng mL^{-1})
Inter-day precision (RSD %)	6.0 (10 ng mL^{-1}) 5.6 (20 ng mL^{-1})	5.8 (50 ng mL^{-1}) 5.9 (100 ng mL^{-1})

[a] Preconcentration factor.

[b] Linear dynamic range (ng mL^{-1}).

[c] Limit of detection (S/N = 3), (ng mL^{-1}).

[d] Relative standard deviation (n = 5).

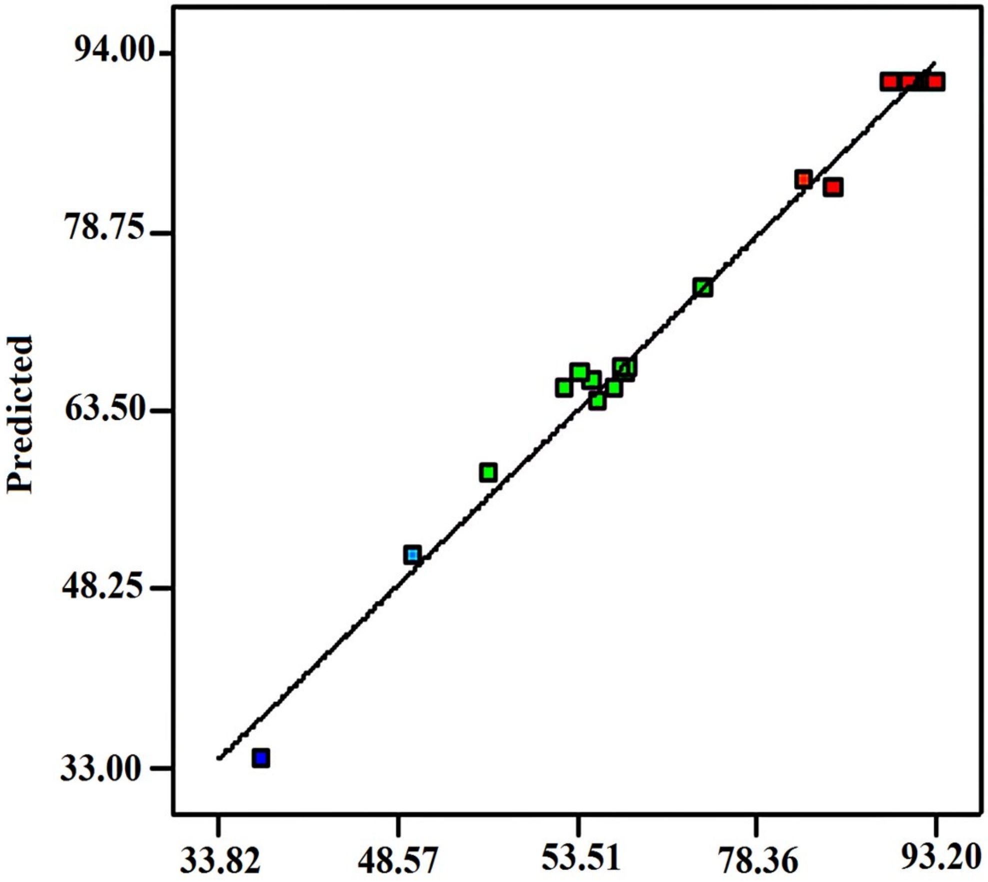

Fig. 1S. Plot of predicted values vs. actual values for the recovery (%) of Cd^{2+} ions in the absorption step.

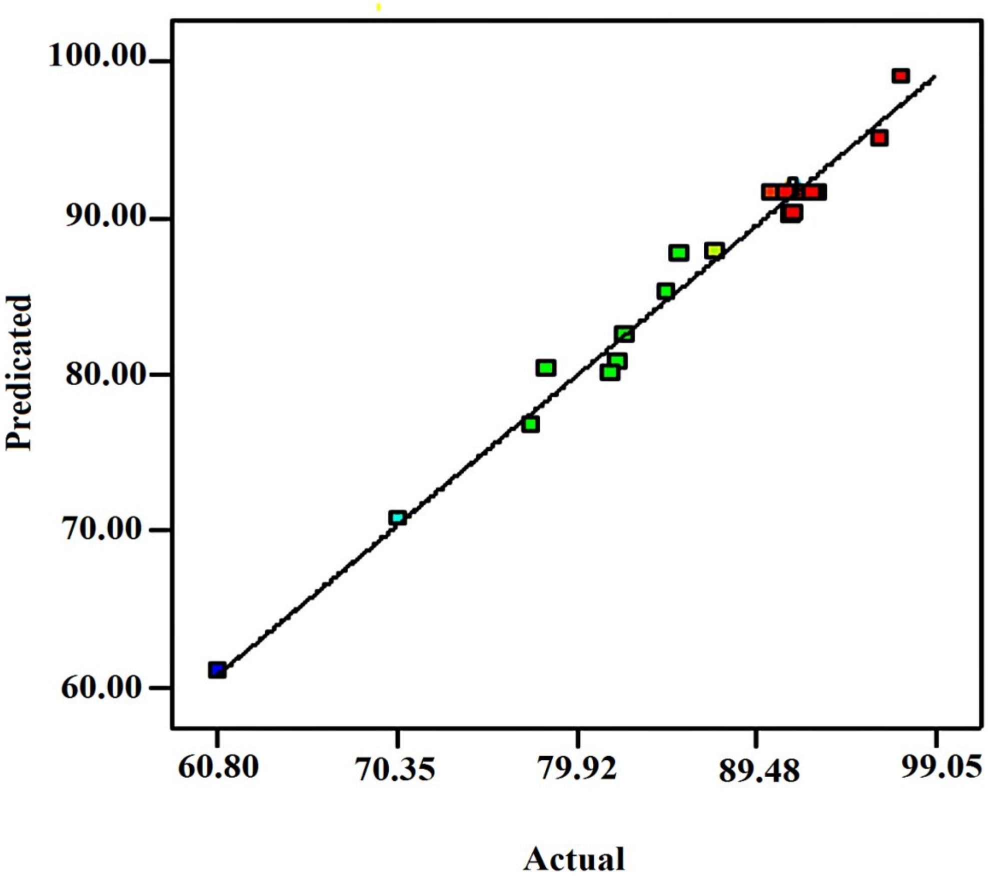

Fig. 2S. Plot of predicted values vs. observed values for the recovery (%) of Pb^{2+} ions in the desorption step.

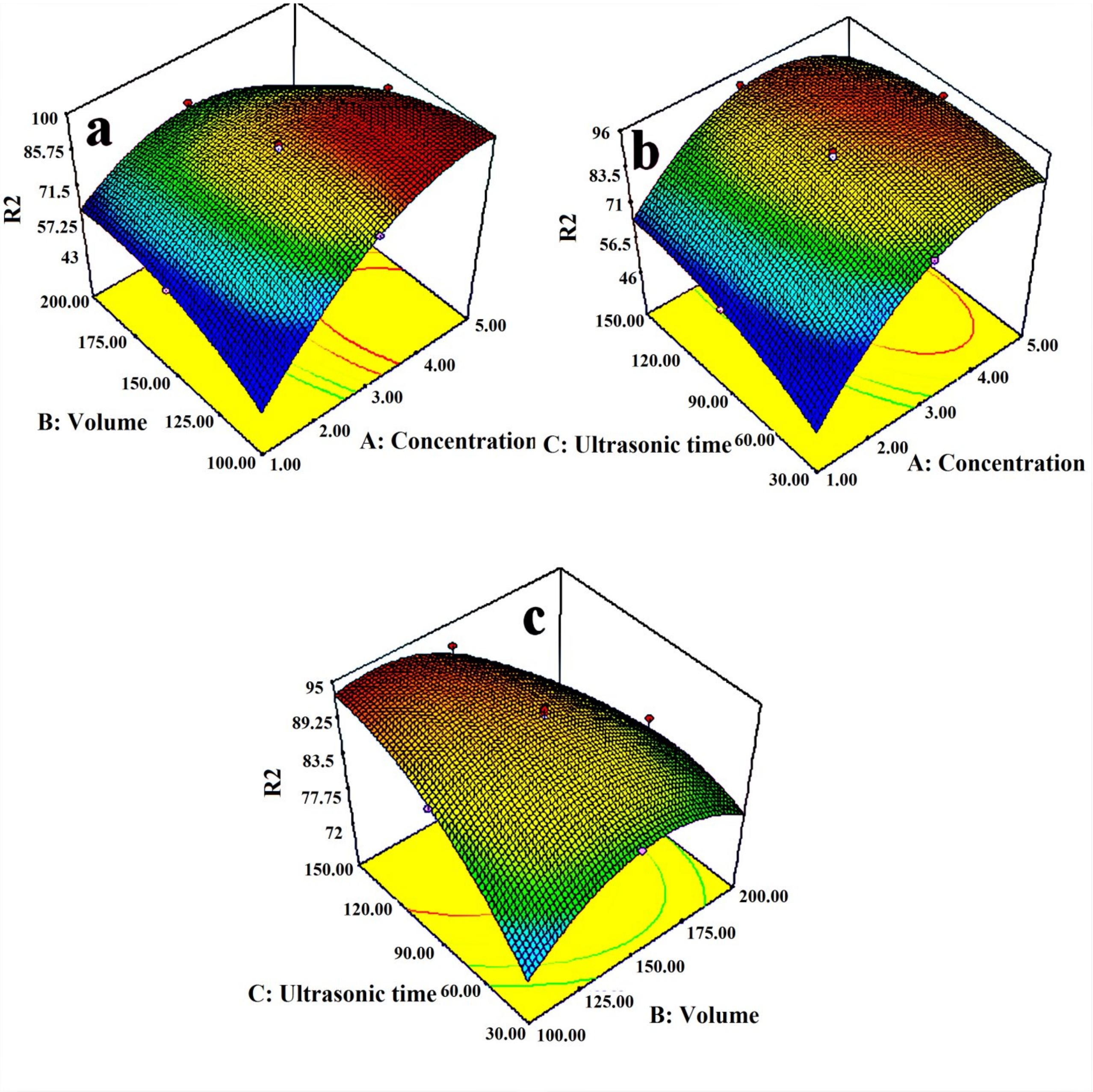

Fig. 3S. Response surfaces for Pb^{2+} as a representative analyte in the desorption step: (a) concentration of eluent vs. volume of eluent; (b) concentration of eluent vs. ultrasonic time; (c) volume of eluent vs. ultrasonic time, respectively.