## **ELECTRONIC SUPPLEMENTARY INFORMATION**

## Surface Engineering: Binary MgFe-LDH·xFe<sub>3</sub>O<sub>4</sub> nanocomposites for Improved Magnetic Solid-Phase Extraction of Pharmaceuticals from Aqueous Solution

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**Figure S1.** UV-Vis spectra of Diclofenac Sodium solution in the full linearity range



*Figure S2.* Calibration curves for determination of Diclofenac Sodium at various wavelengths: 276 nm and 199 nm



**Figure S3.** SEM images of pristine  $Fe_3O_4$  (a),  $Mg_4Fe_-LDHs$  (b),  $Mg_4Fe_-LDH \cdot 0.1Fe_3O_4$  (c),  $Mg_4Fe_-LDH \cdot 0.3Fe_3O_4$  (d),  $Mg_4Fe_-LDH \cdot 0.5Fe_3O_4$  (e) and  $Mg_4Fe_-LDH \cdot 1.0Fe_3O_4$  (f) samples



Figure S4. EDX mapping (a) and spectrum (b) of Mg, Fe-LDHs sample



*Figure S5.* XRD patterns of *Mg,Fe-LDH-0.3Fe<sub>3</sub>O<sub>4</sub>* calcinated at 800°C and corresponding references (MgFe<sub>2</sub>O<sub>4</sub> (# 38997-ICSD) and MgO (# 29127-ICSD))from ICSD database



Figure S6. Speciation diagram of DCF as a function of the pH solution



*Figure S7.* The  $pH_{PZC}$  determination for obtained adsorbents (*Conditions*: weight 0.050 g, volume 50 mL, time 24 h, C(NaClO<sub>4</sub>) = 0.1 M)

*Table S1.* Parameters for the intra-particle diffusion kinetic model of the asprepared materials

Sample	1 <sup>st</sup> stage			2 <sup>nd</sup> stage			3 <sup>rd</sup> stage		
	Ki	С	R <sup>2</sup>	Ki	С	<b>R</b> <sup>2</sup>	Ki	С	<b>R</b> <sup>2</sup>
Mg,Fe-LDHs	0.0463	0.0012	0.9996	0.024	0.134	0.9279	0.00075	0.3781	0.7494
Mg,Fe-LDH·0.3Fe <sub>3</sub> O <sub>4</sub>	0.0407	0.0028	0.9974	0.023	0.109	0.9224	0.0016	0.332	0.8836
Mg,Fe-LDH ·0.5Fe <sub>3</sub> O <sub>4</sub>	0.0295	0.0016	0.9974	0.020	0.062	0.9447	0.0310	0.2392	0.8394
Fe <sub>3</sub> O <sub>4</sub>	0.00265	0.0005	0.9805	-	-	-	0.0009	0.0158	0.9142

Table footnotes.  $K_i$  - rate constant of intraparticle diffusion, (mmol·g<sup>-1</sup> min<sup>-1/2</sup>); C - the intercept, (mmol/g).



**Figure S8.** Adsorption isotherms (mmol/g) of DCF onto *Mg*,*Fe*-*LDHs* and corresponding magnetic nanocomposites at room temperature (*Conditions:*  $pH = 7.5 \pm 0.1$ , m/V = 1.00 g/L, time overnight, at room temperature)





**Figure** *S***9.** Linear fitting of adsorption isotherms with Langmuir (*a*), Freundlish (*b*) and Temkin (c) equations for DCF on obtained samples