Supplement information

Synergistic effect of humic and fulvic acids on Ni removal by the calcined Mg/Al layered double hydroxide

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S1. Characterization method of humic (HA) and fulvic (FA) acids

The elemental compositions of the HA and FA samples was determined on a Vario EL III CHN analyzer (Elementar, Germany). The concentrations of carboxylic acid (-COOH) and phenolic groups (Ar-OH) in the HA and FA samples were determined by potentiometric titration using a Metrohm iTouch 916 (Metrohm Inc, USA) under argon flushing.

The molecular weight distribution (MWD) of the organic matters (HA and FA) in the test water sample was obtained using HPSEC (high-performance size-exclusion chromatography) method, which included a high performance liquid chromatography (HPLC, Waters 1525, USA) coupled with a gel-chromatography column (Shodex Protein KW-802.5, Sholo, Japan). The mobile phase was prepared using 0.02 M orthophosphate and 0.1 M NaCl (pH 6.8), and then, degassed for at least 1 h prior to use. The flow rate of the mobile phase was 0.8 mL/min. The DOM sample was filtered through a 0.22 μm membrane filter before analysis on HPSEC. Polystyrene sulfonate (PSS) with molecular weight (MW) ranging from 1800 Da to 35,000 Da in acetone (MW = 58 Da), was used as standards to quantify the fractions of natural organic matters with different MWs. The UV absorbance of PSS and acetone was monitored at 224 nm. When DOM was analyzed, the sample was taken and filtered through a 0.22 μm membrane filter for HPSEC analysis. The UV absorbance of the sample was monitored at 254 nm.

S2. Zeta potential determination method of Mg/Al LDH

Zeta potential of the LDH suspension at pH ranging from 4 to 12 was determined by using a Zetasizer Nano ZS (Malvern, England). Briefly, 10 mg of the LDH were dispersed in 50 mL Milli-Q water, and then the pH was adjusted by using 0.1 M of HNO_3 or NaOH to achieve desired pH values. This experiment was carried out in triplicates.

S3. **The distribution of Ni vs pH**

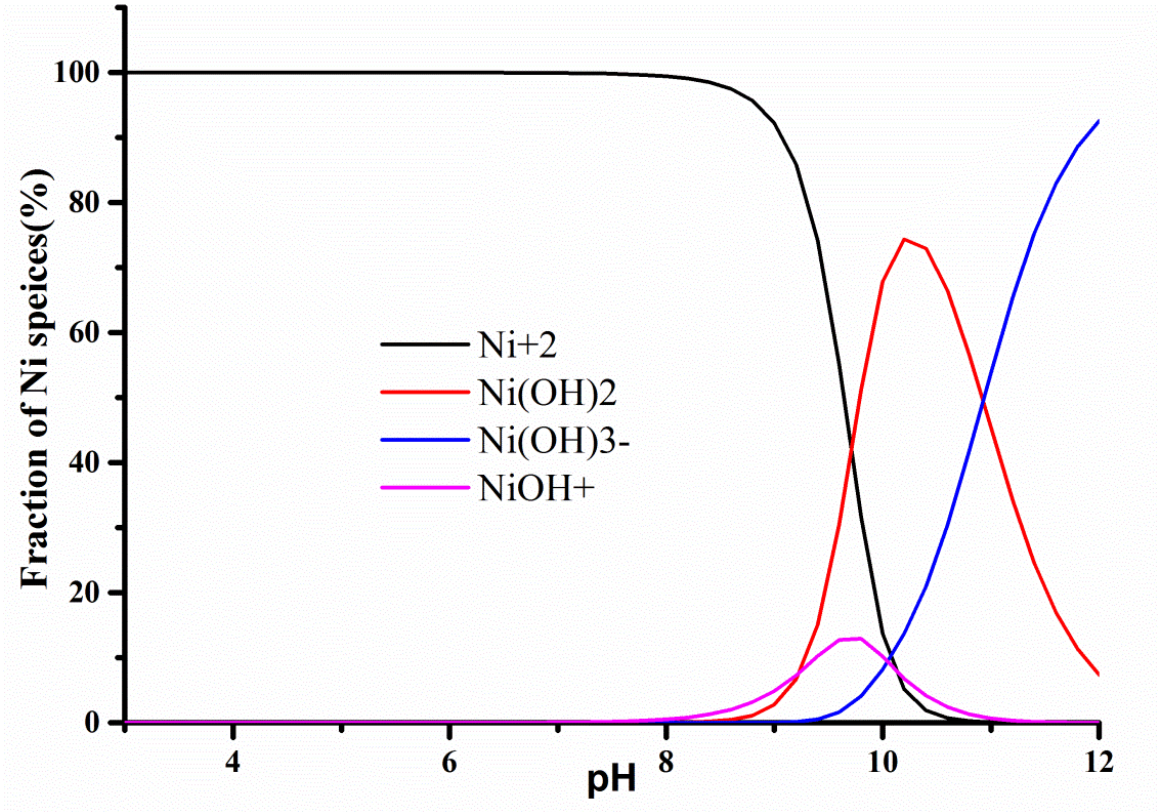

Fig. S1. The distribution of Ni species as a function of pH simulated by using visual Minteq. ver. 3.0

S4. Zeta potential of Mg/Al LDH vs pH

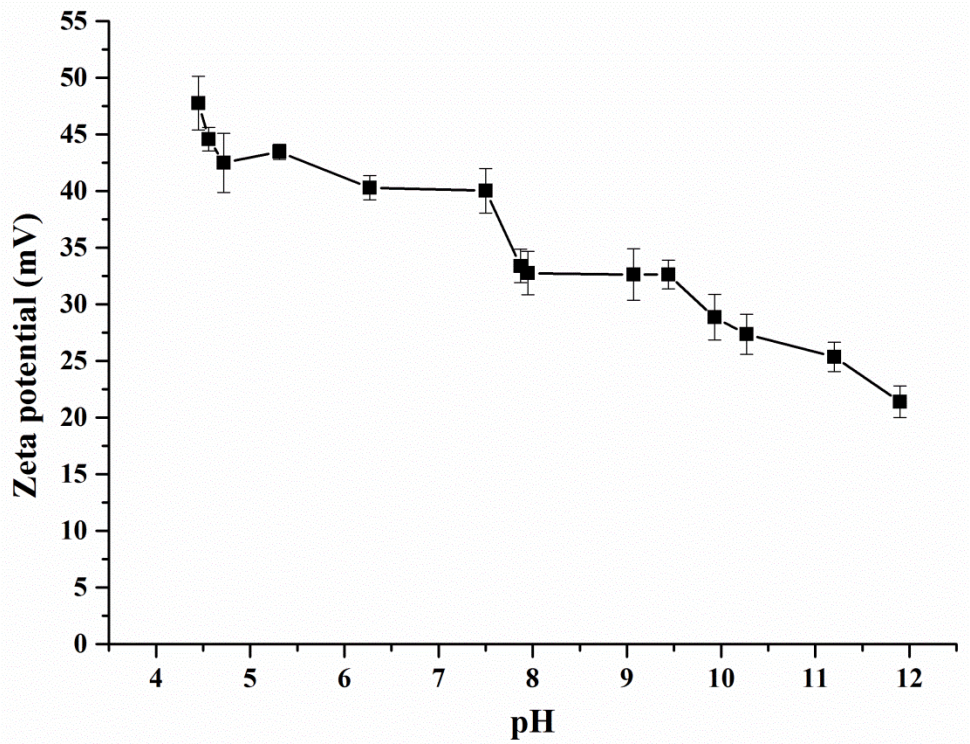

Fig. S2. Zeta potential of Mg/Al LDH as a function of pH. Samples at each point were performed in triplicates.

S5. Adsorption isotherm of Ni(II) by Mg/Al LDH at pH 7

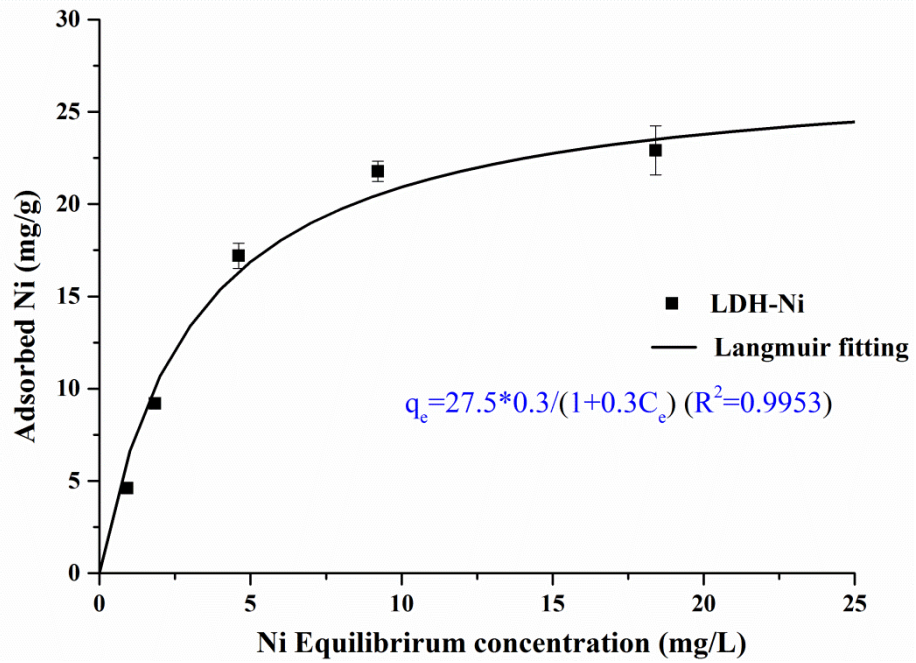

Figure S3. Adsorption isotherm of Ni by Mg/Al LDH at pH 7.

S6. Characteristics of HA and FA

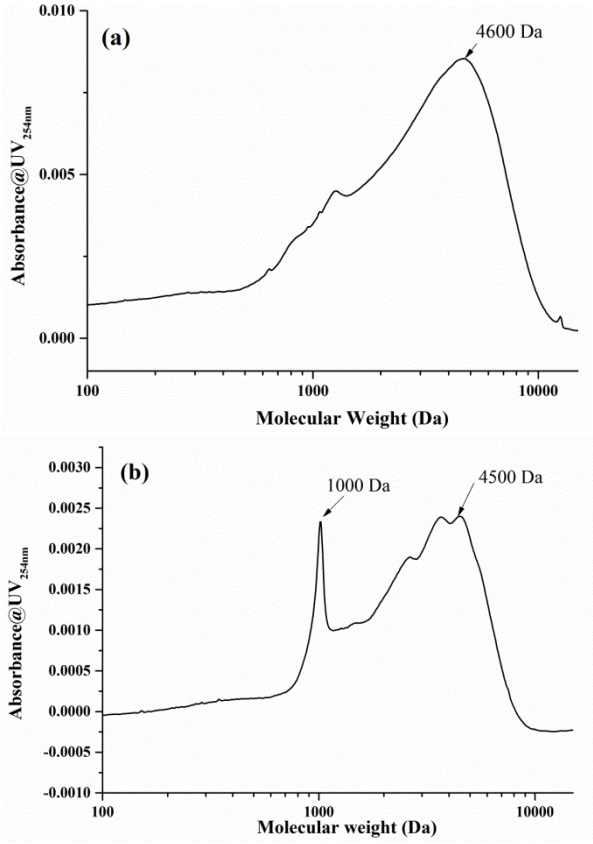

Fig. S4. Molecular weight distribution of HA (a), and FA (b)

Table S1. Selected physicochemical properties of humic (HA) and fulvic acids (FA) samples in this study[a]

Organic matter	Elemental compositions (molar %)				Functional groups (mmol/g)	
	C	H	N	O	Carboxyl groups	Phenol groups
HA	54.5 ± 0.2	5.7 ± 0.1	0.8 ± 0.1	39.0 ± 0.3	4.21 ± 0.05	2.10 ± 0.03
FA	53.1 ± 0.1	8.1 ± 0.1	0.5 ± 0.1	38.3 ± 0.2	5.65 ± 0.01	1.87 ± 0.02

[a] Average value ± standard deviation (n=3);

S7. Selected elemental analysis of the calcined LDH before and after reaction with Ni ions

Table S2. The selected elemental analysis of the calcined LDH before and after reaction with Ni[a]

	Elemental contents in solid (mg/g)			Atomic content (mol/g)		
	Mg	Al	Ni	Mg	Al	Ni
Calcined LDH	234.5 ± 1.5[b]	85.1 ± 2.2	NA[c]	9.8 ± 0.1	3.2 ± 0.1	NA
Calcined LDH-Ni	140.7 ± 2.1	84.5 ± 1.3	210.0 ± 5.4	5.9 ± 0.1	3.1 ± 0.1	3.6 ± 0.1

[a] The solid content in the solution was 0.2 g/L, the initial concentration of Ni (II) was 120 mg/L. the pH of the reaction remained at pH 7.0; the measurement was played in triplicates.
[b] average value ± standard deviation (n=3)
[c] NA: not available.

S8. Stainless steel wastewater composition

The performance of the calcined LDH in practical application was further tested. The wastewater was sampled in the effluent of the 2nd sedimentation tank (before the coagulation tank) of the wastewater treatment plant from one of the Word largest stainless steel manufactory locating in Shiyan, Hubei province in China.

Table S3. The major chemical composition of the wastewater (unit: mg/L except for pH)

Parameters	pH	Ni^{2+}	Total Fe	$Cr_2O_7^{2-}$	F^-	SS	Cd^{2+}	NO_3^-
Before treatment	6.0	530	675	8.5	1200	321	12.3	1420