

Supporting information for

Three Dimensional Nanowall of Calcein/Layered Double Hydroxide: Towards Electrogenerated Chemiluminescence Sensor

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Experimental section

1.1 Preparation of the parallel Calcein/LDH film

The Mg₂Al-NO₃ LDH precursor was synthesized by the hydrothermal method reported previously. Subsequently, the Calcein intercalated LDH composites were prepared following the ion-exchange method. Calcein (*a* mol) were dissolved in 150 mL of water, in which $x = a/n(\text{Al}) = 0.1\%, 1.25\%, 10\%, \text{ and } 100\%$, respectively. This solution was adjusted to pH=7.0 with a NaOH (0.2 mol/L) solution. Then freshly prepared Mg₂Al-NO₃ LDH (0.5 g) was dispersed in the solution thoroughly. The suspension was adjusted to pH=7.0 and stirred at room temperature under N₂ atmosphere for 48 h. The product Calcein/LDH (*x*%) was washed extensively with water.

1.2 Fabrication of the parallel Calcein/LDH (*x*%) films

The parallel films of Calcein/LDH (*x*%) were fabricated by solvent evaporation method. Substrates of ITO wafer were firstly cleaned by immersing in a bath of deionized water in an

ultrasonic bath for 30 min. Pasty Calcein/LDH ($x\%$) (0.05 g) was suspended in 20 mL of water under N_2 atmosphere in an ultrasonic bath (99 W, 28 kHz) at room temperature for 5 min. Then, the resulting Calcein/LDH ($x\%$) suspension was dropped on quartz and ITO substrates and dried in vacuum at ambient temperature for 5 h.

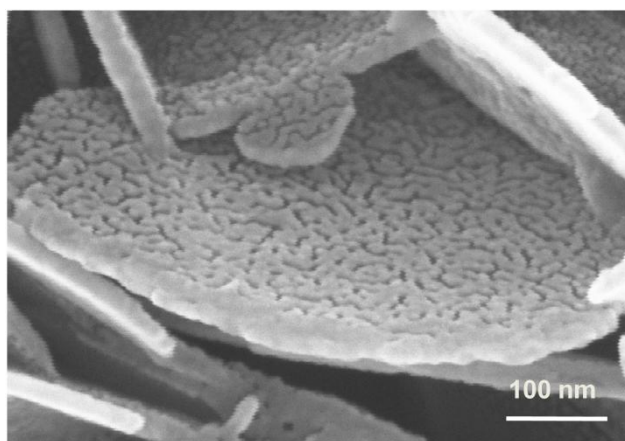


Figure S1. Top-view SEM images of the Calcein/LDH nanowall film under high-magnification.

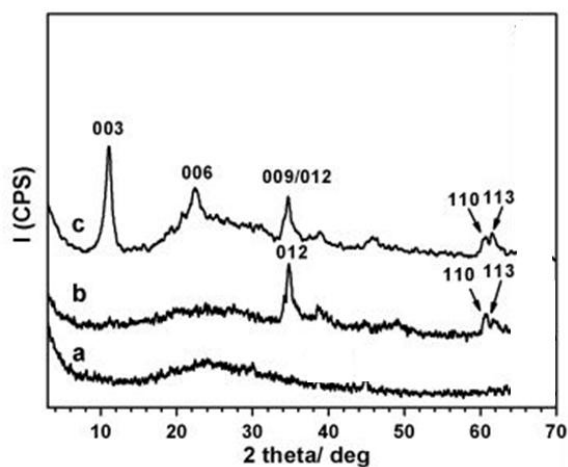


Figure S2. XRD patterns of (a) ITO substrate, (b) the $MgAl-CO_3-LDHs$ film, and (c) the $MgAl-CO_3-LDHs$ powder scraped from the film.

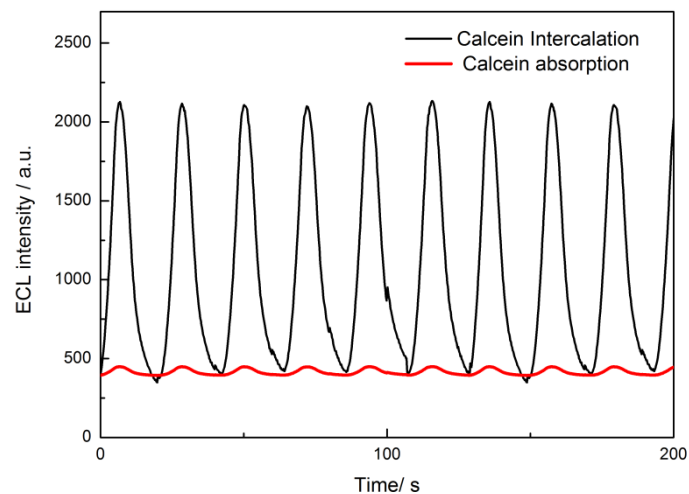


Figure S3. ECL spectra of the Calcein/LDH film and Calcein adsorbed LDH film.

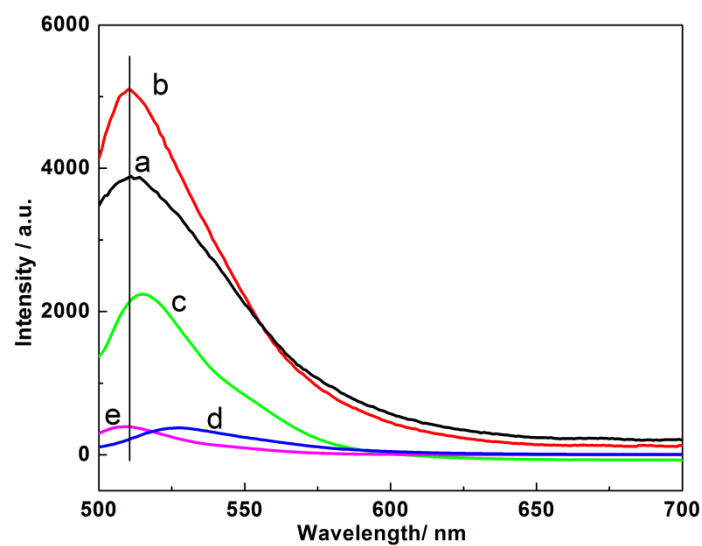


Figure S4. (A) The photoemission spectra of the parallel Calcein/LDH ($x\%$) film for (a)~(d) $x = 0.1\%$, 1.25% , 10% , and 100% , respectively and (e) pristine Calcein in solution with the excitation wavelength of 490 nm .

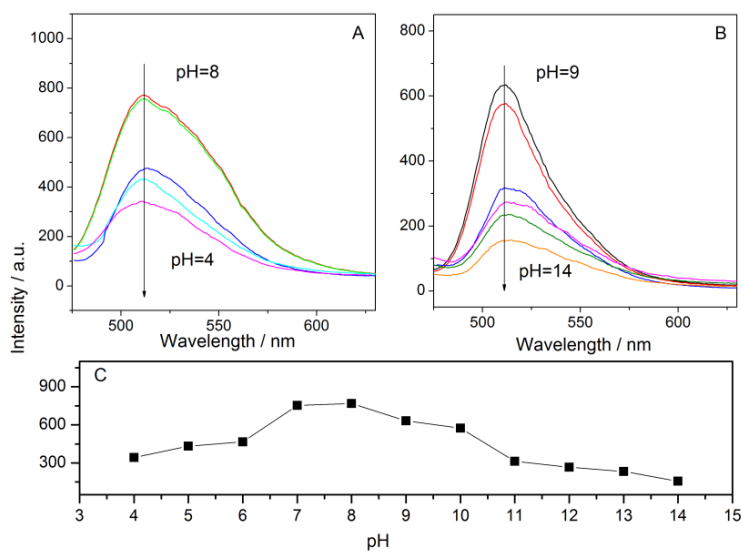


Figure S5. (A) and (B) Emission spectra of the Calcein/LDH nanowall film at different pH values (295 K, $\lambda_{\text{ex}}=498$ nm); (C) pH titration curve for the Calcein/LDH nanowall film.

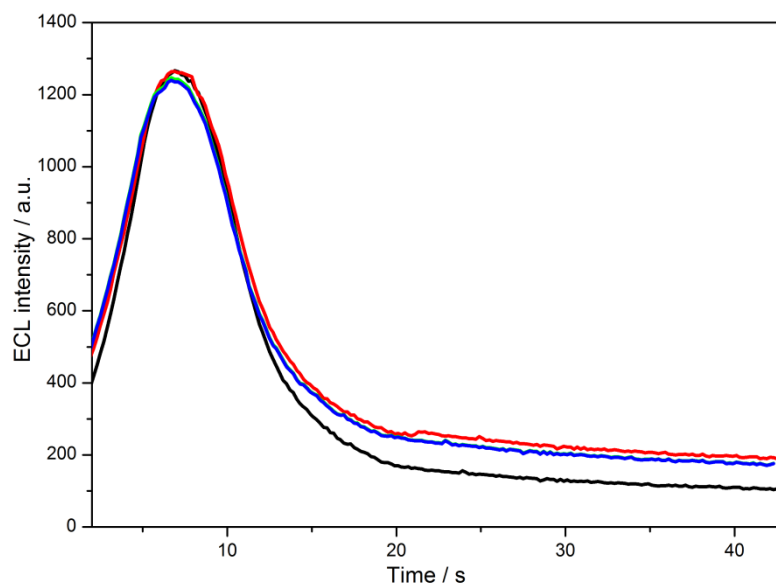


Figure S6. Storage stability of ECL sensor for the Calcein/LDH nanowall film stored in 0.1 M PBS of pH 7.4 at 4 °C for one month.

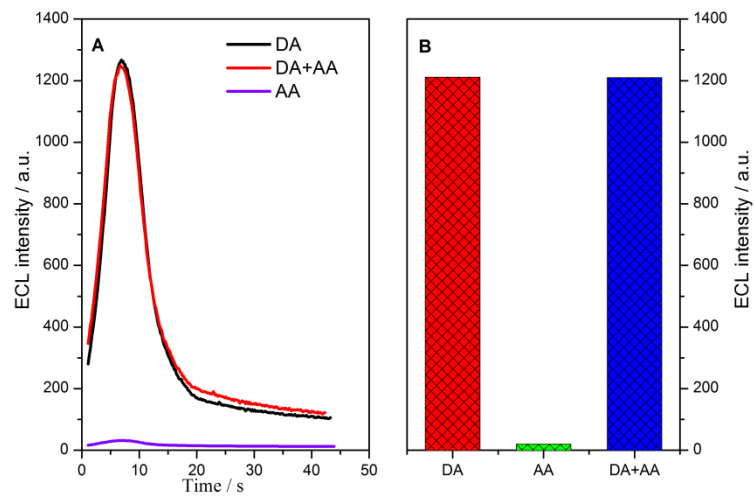


Figure S7. (A) ECL spectra of the Calcein/LDH nanowall film and (B) column graph recorded in 0.1 mM DA and 50 mM AA (0.1 M PBS at pH=7.4).