Electrochemical deposition to construct nature inspired multilayer chitosan/layered double hydroxides hybrid gel for stimuli responsive release of protein

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Figure S1. SEM images of NO₃-LDHs (A) and CO₃-LDHs (B).



Figure S2. SEM images of chitosan/INS-LDHs hydrogels with mass ratios of: (A) 3:1; (B) 3:4; (C) 3:5; (D) 1:2.



Figure S3. Cumulative release profiles of insulin from chitosan/INS-LDHs hydrogel in 0.9% NaCl solution. The release was performed under different pHs (4.0, 7.0, 9.0) with 0 V voltage applied.

The preparation of NO₃-LDHs and CO₃-LDHs:

 NO_3 -LDHs was prepared according to a published literature with minor modification.¹ $Mg(NO_3)_2 \cdot 6H_2O$ (15.39g), AI (NO_3)_2 $\cdot 9H_2O$ (11.26g) and $NaNO_3$ (4.25g) were added and dissolved in 280 ml water under N_2 atmosphere. Then diluted ammonia water (6 wt %) was added to the solution at a speed of 25 ml/min till the final pH 9.5. The precipitate was aged for 1.5 h at room temperature and then washed with deionized water. After that, the filter cake was peptized at a constant temperature of 80 °C, formed the positive sol, and dried at 65 °C to get NO_3 -LDHs.

CO₃-LDHs was prepared according to a published literature.² Mg(NO₃)₂·6H₂O (19.2g) and Al(NO₃)₃·9H₂O (9.38g) was dissolved in water and this solution was added drop-wise to a basic solution (100 ml) containing 0.05 mol Na₂CO₃. The pH value of the basic solution was kept constant at 11 by adding NaOH solution (3.4 M). The resulting mixture was aged at room temperature for 24 h with continuous stirring. The aged mixture was filtered and washed with deionized water until pH = 7, followed by drying at 100 °C in an oven.

References:

- 1. Q. Z. Yang, D. J. Sun, C. G. Zhang, X. J. Wang and W. A. Zhao, *Langmuir*, 2003, **19**, 5570-5574.
- 2. Q. Wang, H. T. Hui, Z. Guo, L. Chen, Y. Liu, J. Chang, Z. Zhong, J. Luo and A. Borgna, *Applied Clay Science*, 2012, **55**, 18–26.