Supporting Information

Fluoride sensing performance of fluorescent NH$_2$-MIL-53(Al):

2D Nanosheets vs. 3D Bulk

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Fig. S1 (a) SEM image of NM5A-NS; (b) Thickness distribution of NM5A-NS measured by counting particles in SEM image; (c, d) TEM image of isolated nanosheets from NM5A-NS obtained by ultrasonication; (e) the Tyndall effect of NM5A-NS; (f) SEM image of NM5A-Bulk.

Fig. S2 SEM images of (a) NM5A-NS; (b) NM5A-NS after alkali treatment (pH = 12) for 1 day.
Fig. S3 The ammonia nitrogen content in the filtrate measured by a spectrophotometer after NM5A-NS immersed in alkaline solutions for 1 day.

Fig. S4 (a) TG curves of NM5A-NS; (b) XRD patterns of NM5A-NS after soaking in different solvents for 3 days.
**Fig. S5** The corresponding fluorescent image of NM5A-NS in different ionic solutions under irradiation of 300 nm lamp.

**Fig. S6** SEM images of NM5A-NS after immersed in fluoride solutions of different concentrations for 1 day (a) 0 µM; (b) 10 µM; (c) 50 µM; (d) 100 µM; (e) 250 µM; (f) 500 µM.
**Fig. S7** XRD patterns of **NM5A-NS** after immersed in fluoride solutions of different concentrations for 1 day (0-500 µM).

**Fig. S8** The ammonia nitrogen content in the filtrate measured by a spectrophotometer after **NM5A-NS** immersed in fluoride solutions for 1 day (10-500 µM).
**Fig. S9** FT-IR spectra of NM5A-NS before and after fluoride treatment.

**Fig. S10** UV-vis spectra of NM5A-NS in various concentrations of fluoride solutions (0-1000 µM).