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3	Fluoride sensing performance of fluorescent NH <sub>2</sub> -MIL-53(Al):
4	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.



9 Fig. S2 SEM images of (a) NM5A-NS; (b) NM5A-NS after alkali treatment (pH = 12)
10 for 1 day.



2 Fig. S3 The ammonia nitrogen content in the filtrate measured by a
3 spectrophotometer after NM5A-NS immersed in alkaline solutions for 1 day.



7 Fig. S4 (a) TG curves of NM5A-NS; (b) XRD patterns of NM5A-NS after soaking in
8 different solvents for 3 days.



2 Fig. S5 The corresponding fluorescent image of NM5A-NS in different ionic

3 solutions under irritation of 300 nm lamp.

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7 Fig. S6 SEM images of NM5A-NS after immersed in fluoride solutions of different

- 8 concentrations for 1 day (a) 0  $\mu$ M; (b) 10  $\mu$ M; (c) 50  $\mu$ M; (d) 100  $\mu$ M; (e) 250  $\mu$ M; (f)
- 9 500 μM.
- 10



- 2 Fig. S7 XRD patterns of NM5A-NS after immersed in fluoride solutions of different
- 3 concentrations for 1 day (0-500  $\mu$ M).



7 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 9  $\mu$ M).





6 Fig. S10 UV-vis spectra of NM5A-NS in various concentrations of fluoride solutions

<sup>7 (0-1000</sup> μM).