

## **Layered NbOCl<sub>2</sub> kinetic degradation mechanism and improved second-order nonlinear optical responses**

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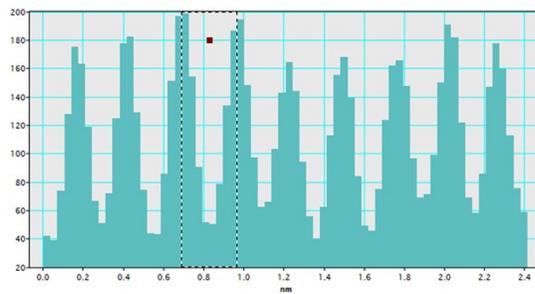


Figure S1: contrast profiles corresponding to the zone in Figure 1d.

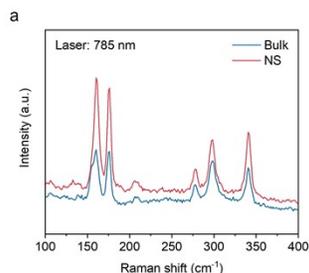


Figure S2: Raman spectra of NbOCl<sub>2</sub> with bulk morphology and nanosheet structure (Laser: 785 nm).

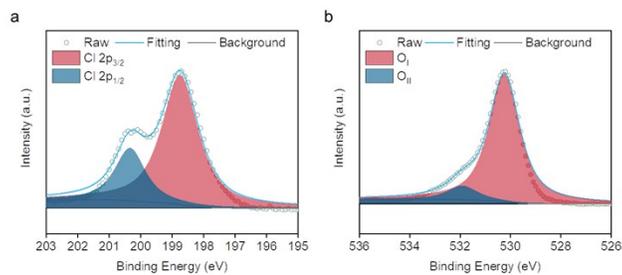


Figure S3: (a) and (b) represent XPS spectra of Cl 2p and O 1s.

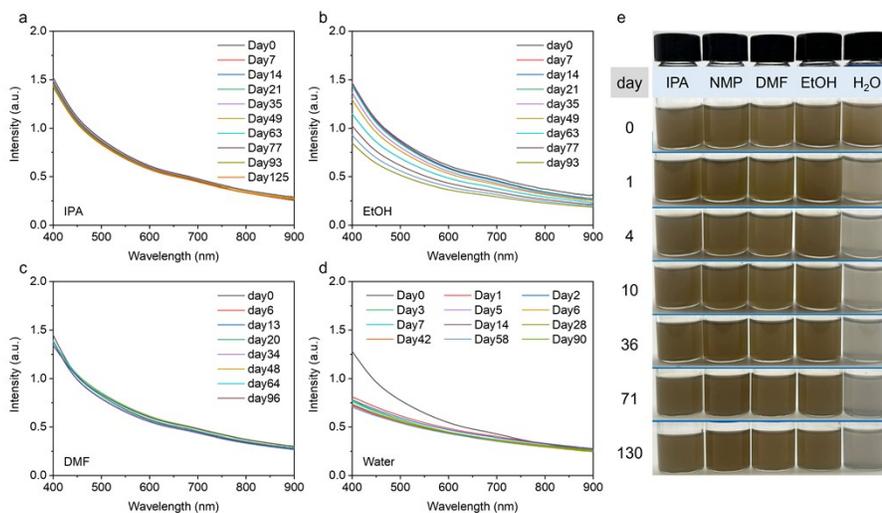


Figure S4: (a)-(d) Extinction spectra of NbOCl<sub>2</sub> nanosheets dispersed in different solvents after storage at 4 °C for a series of times. (e) Optical images of NbOCl<sub>2</sub> nanosheets dispersed in different solvents after being stored under sealed conditions at 4 °C for a series of times.

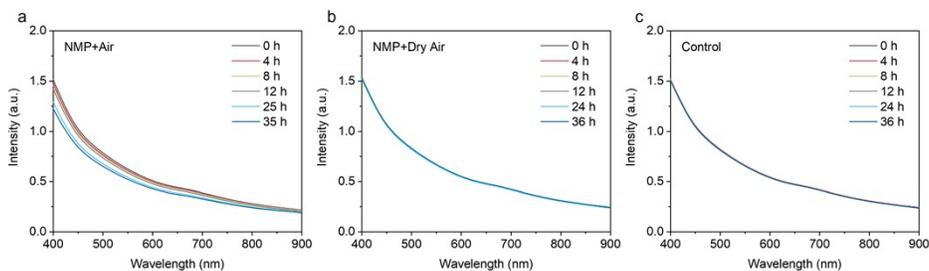


Figure S5: Extinction spectra of NbOCl<sub>2</sub> nanosheets dispersed in NMP with different conditions for a series of times. (a) Introducing air. (b) Introducing dry air. (c) Control group.

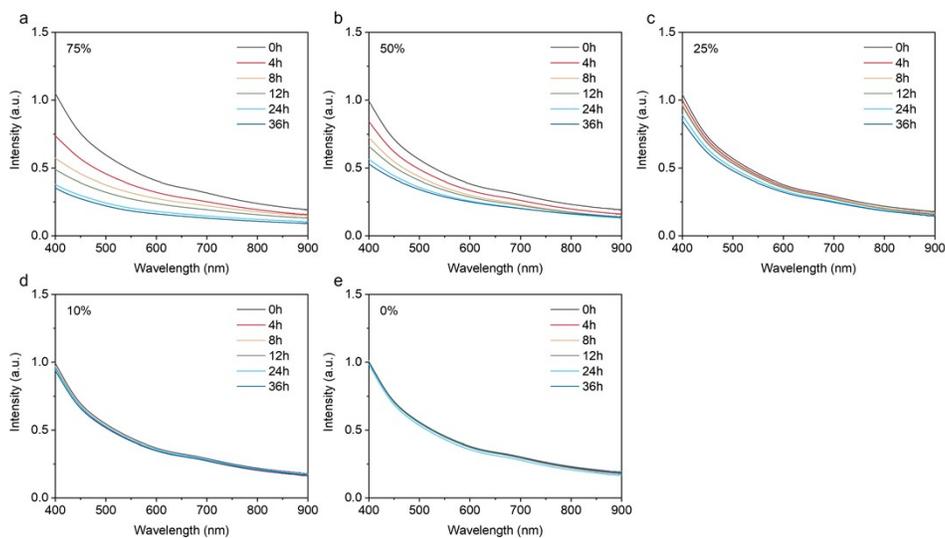


Figure S6: Extinction spectra of NbOCl<sub>2</sub> nanosheets dispersed in NMP/Water mixed solvents with different Water content ratios for a series of times. (a) 75%. (b) 50%. (c) 25%. (d) 10%. (e) 0%.

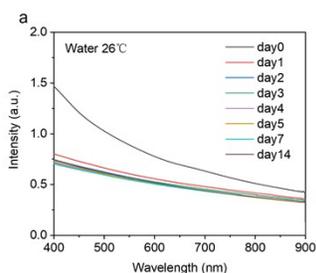


Figure S7: Extinction spectra of NbOCl<sub>2</sub> nanosheets dispersed in water after storage at 26 °C for a series of times.

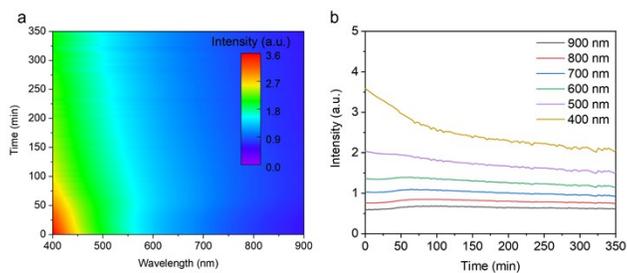


Figure S8: (a) 2D color extinction spectra mapping of NbOCl<sub>2</sub> nanosheet with degradation under uniform dispersion. (b) 2D color first derivatives mapping from (a). (c) Intensity values at different wavelengths are extracted from (a) as a function of time.

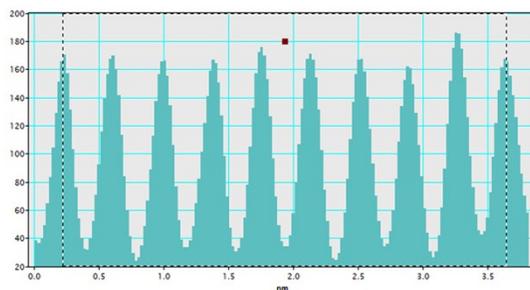


Figure S9: Contrast profiles corresponding to the zone in Figure 3a.

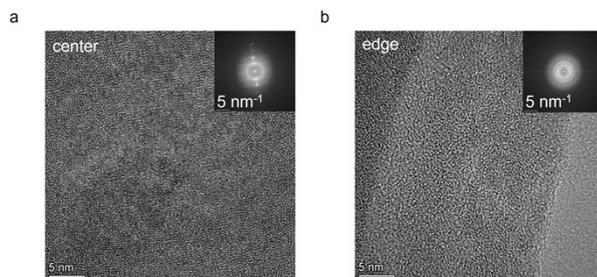


Figure S10: (a) and (b) are HRTEM images from Figure 3b corresponding to the center and edge of nanosheets. The inserted photo is the corresponding SEAD.

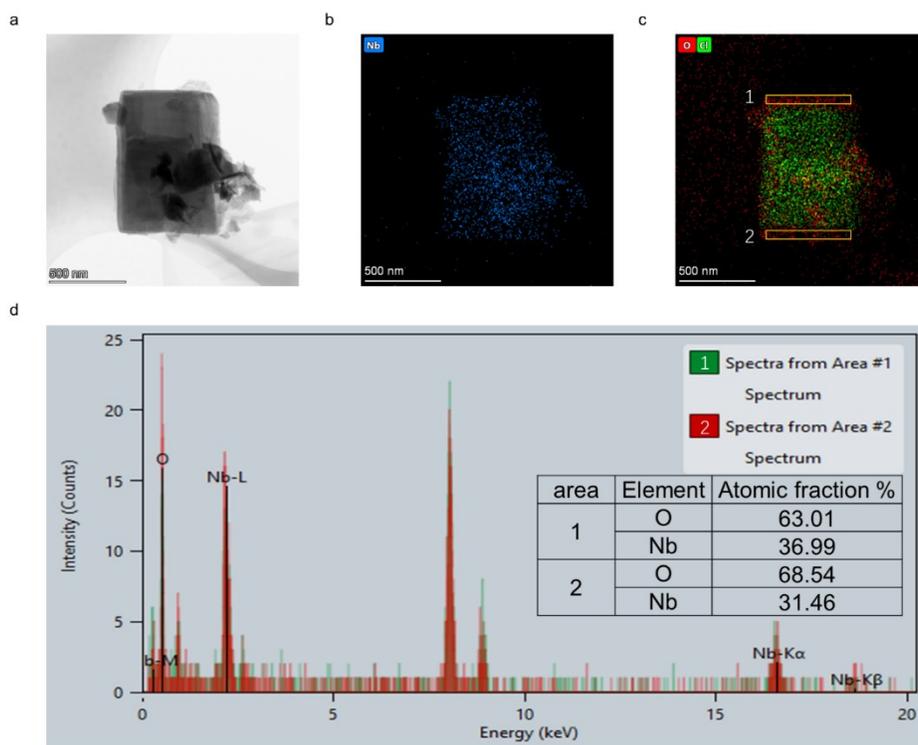


Figure S11: (a) TEM images depicting the morphology of samples degraded for 2 hours followed by annealing. (b) and (c) correspond to EDS-mapping of (a). (d) Energy spectra of Nb and O elements in the marked region of (c).

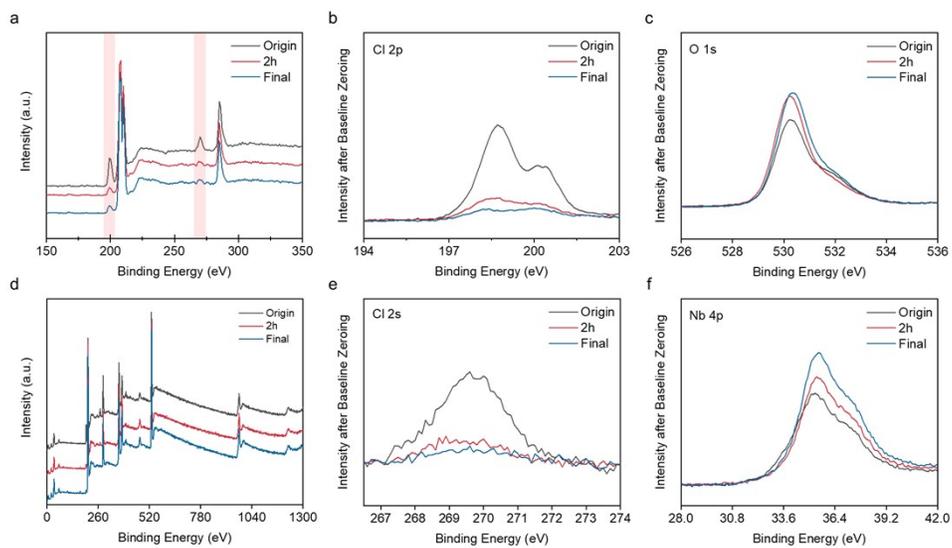


Figure S12: [Cl 2p (b), O 1s (c)] and [Cl 2s (e), Nb 4p (f)] are the narrow scan spectra in the XPS survey spectrum (a) and (d) respectively.

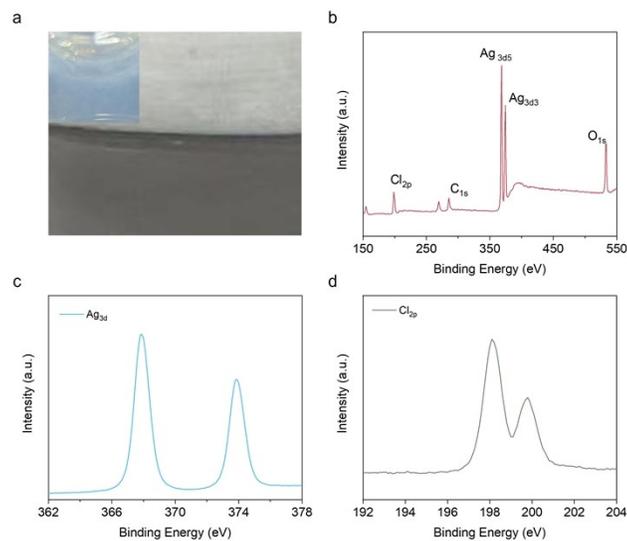


Figure S13: (a) Image of the sediment after it has been stored for a long time. Insert: image of  $\text{AgNO}_3$  being dropped into the reaction solution. (b) XPS survey spectrum of sediment in (a). (c) and (d) are narrow scan spectra of Ag 3d and Cl 2p respectively.

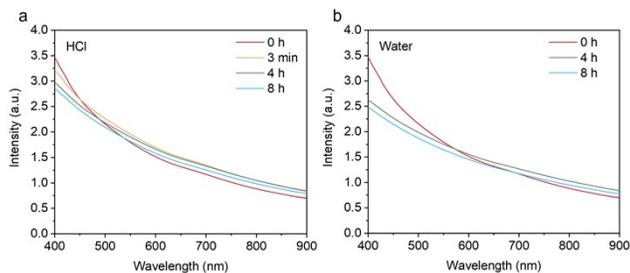


Figure S14: Extinction spectra of  $\text{NbOCl}_2$  nanosheets dispersed in the different environments for a series of times. (a) water. (b) strong acid.

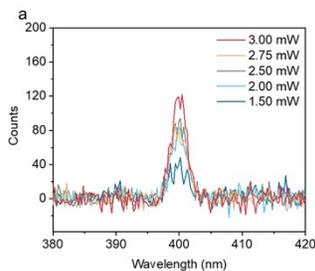


Figure S15: Excitation power dependence SHG spectrum of Final (collection time: 5s).