Electronic Supplementary Information for

A one-pot method to grow pyrochlore $H_4Nb_2O_7$-octahedron-based photocatalyst

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1. Experimental part and photocatalytic test

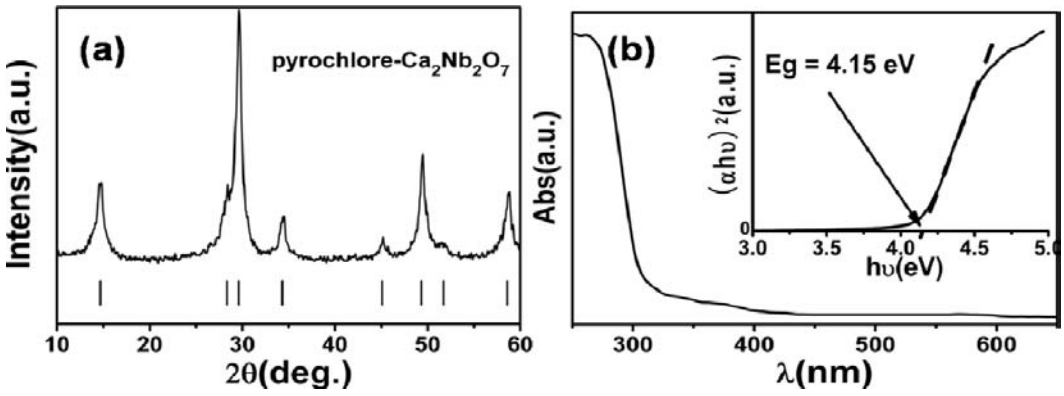

Fig. S1 (a) XRD pattern for the obtained $Ca_2Nb_2O_7$. The Brag peaks position (JCPDS 73-0597) was shown in bottom and (b) UV-Vis spectra with band gap in the inset

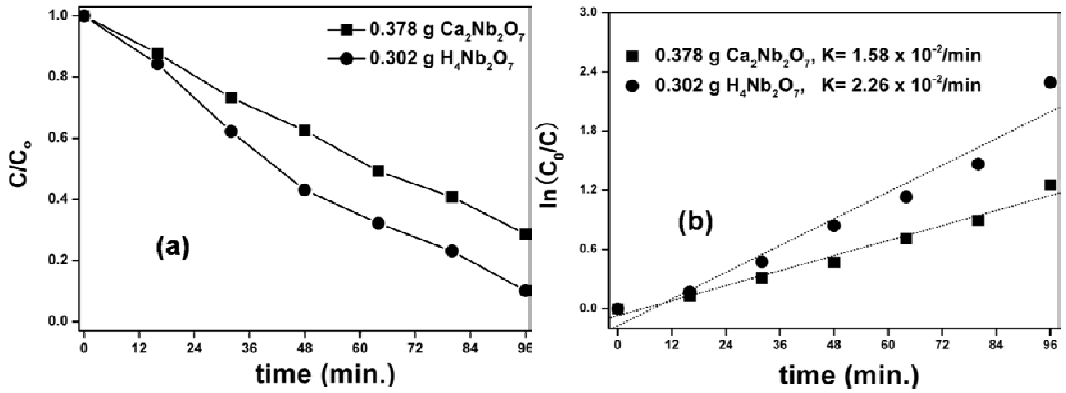

Fig. S2 MO photocatalytic (a) decolorization, (b) kinetics over $Ca_2Nb_2O_7$ and $H_4Nb_2O_7$ with the same molar amount of niobium

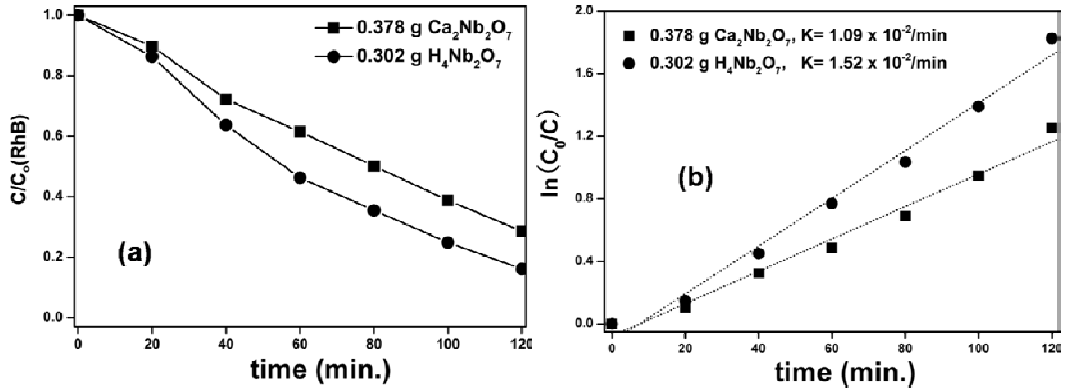

Fig. S3 RhB photocatalytic (a) decolorization, (b) kinetics over $Ca_2Nb_2O_7$ and $H_4Nb_2O_7$ with the same molar amount of niobium

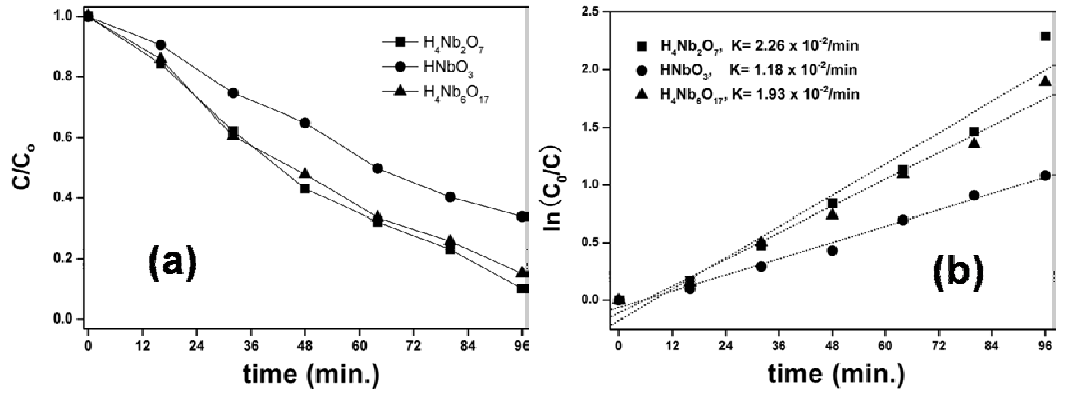

Fig. S4 Photocatalytic test results for $HNbO_3$ and $H_4Nb_6O_{17}$

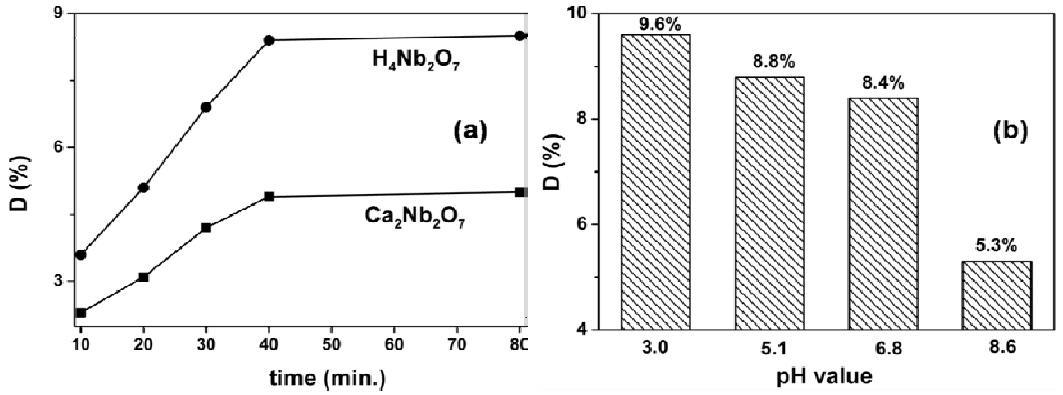

Fig. S5 Equilibrium adsorption of MO. D = $(C_0 - C)/C_0$ * 100%, where C_0 is the initial MO concentration and C is the concentration after equilibrium adsorption. (a) Variation of the MO concentration in the dark over $Ca_2Nb_2O_7$ and $H_4Nb_2O_7$, (b) pH-dependent adsorption of MO over $H_4Nb_2O_7$

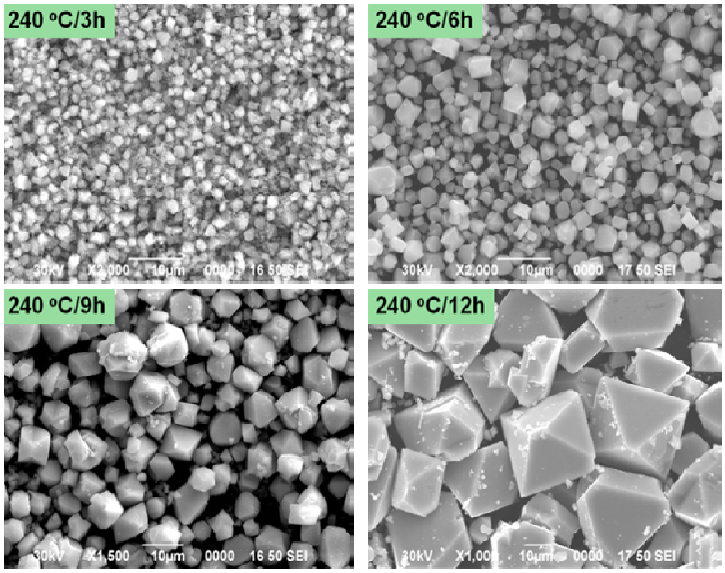

Fig. S6 SEM images for the obtained pyrochlore niobic acids at various reaction time

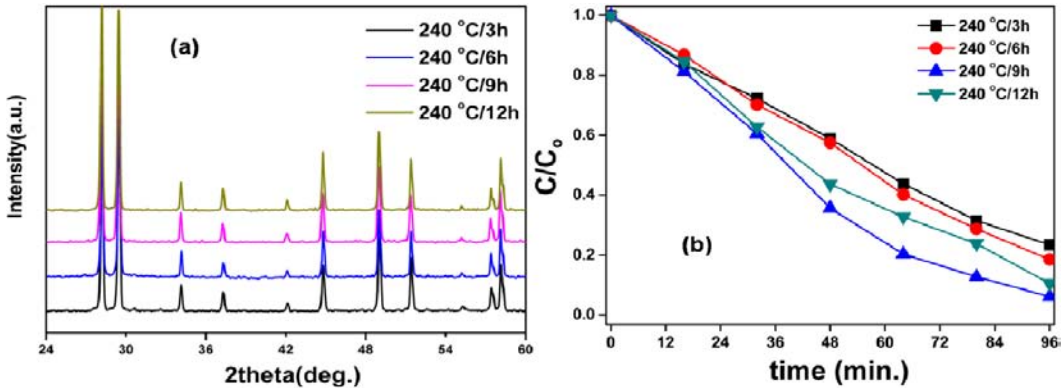

Fig. S7 (a) XRD and (b) photocatalytic test of MO over these niobic acids

Table S1 S_{BET} and reaction constant K of the niobic acid

Catalyst	S_{BET}(m^2/g)	K(10^{-2}/min)	R
240 ^oC/3h	6.2	1.52	0.987
240 ^oC/6h	4.6	1.74	0.984
240 ^oC/9h	3.8	2.93	0.986
240 ^oC/12h	1.2	2.21	0.980

2. Refinement information:

Table S2 Structural Information for the niobic acid

Sample	The obtained pyrochlore niobic acid
Formula	$H_4Nb_2O_7$, Z = 8
Formula weight	301.8 g/mol
Space group	*Fd-3m*(227)
Cell parameters	a = 10.5690(3)Å, α = 90 ^{o}
Cell volume	1180.59(3) $Å^3$
Calc. density	3.3507(1) g/cm^3
Refined parameters*	R_p= 0.0968, R_{wp} = 0.1377

* $R_p = \sum(y_i-y_{ci})/\sum y_i$, $R_{wp} = [\sum w_i(y_i-y_{ci})^2/\sum(w_i y_i^2)]^{1/2}$. Where, y_i and y_{ci} are the observed and calculated intensities, w_i is the weighting factor, n is the total number of y_i data when the background is refined, and p is the number of adjusted parameters. Rietveld refinements were performed with EXPGUI and GSAS program packages [1, 2] using $H_4Ta_2O_7$ as starting model. After refining the scale factor, the terms for background function, unit cell parameters, zero point error, profile coefficients, atomic positions, and isotropic displacement parameters, the (111) and (222) peaks show the largest discrepancy, indicating that the structure of niobic acid crystal is {111} oriented. The M.D. model was applied for the preferred orientation with the

refined r = 0.72. The detailed refinement results are presented in the following Table S2 & S3.

Table S3 Atomic parameters for the niobic acid

Atom	Wyck.	site	x/a	y/b	z/c	U(Å^2)
Nb1	16d	-3m	0.5	0.5	0.5	0.0366(2)
O1	48f	2mm	0.436	0.125	0.125	0.023(5)
O2	8a	-43m	0.125	0.125	0.125	0.023(5)

Table S4 The bond and distance data for the NbO_6 octahedron

Atom 1	Atom 2	Atom 3	d $_{1, 3}$ (nm)	angle $_{2, 1, 3}$ (deg.)
O1	Nb1	Nb1	0.19870	140.196
Nb1	O1	O1	0.19870	90.608
Nb1	O1	O1	0.19870	180.00
Nb1	O1	O1	0.19870	89.392

3. Packing factor (PF)

The packing factor (PF) is defined as follows,

$$PF = Z\,(xV_A + yV_B + zV_C) / V_{cell},$$

where, Z is the number of the formula unit in one unit cell of a semiconductor ($A_xB_yC_z$), V_A, V_B and V_C are ion volumes calculated by assuming spherical ions with a Shannon radius that depends on the coordination number, and V_{cell} is the cell volume. The structural parameters and the calculated PF are listed in Table S5.

Table S5. Packing factor (PF) of $H_4Nb_2O_7$ and $Ca_2Nb_2O_7$

Compound	$H_4Nb_2O_7$	$Ca_2Nb_2O_7$
Space group	F*d-3m* (227)	F*d-3m* (227)
Cell parameters	a = 10.5690Å, Z = 8	a = 10.4445Å, Z = 8
V_{cell}	1180.60 $Å^3$	1139.4 $Å^3$
PF	47.56%	54.99%

References

S1 A. C. Larson, R.B. Von Dreele, "General Structure Analysis System (GSAS)", Los Alamos National Laboratory Report LAUR 86–748 (1994).

S2 B. H. Toby, EXPGUI, A graphical user interface for GSAS, *J. Appl. Cryst.,* 2001, **34**, 210.

S3 X. P. Lin, J. J. Wu, and F. Q. Huang, *Phys. Chem. Chem. Phys.,* 2009*,* **11***,* 10047.