Electronic Supplementary Information (ESI)

New-phased fluorine-doped $H_2(H_2O)Nb_2O_6$ photocatalyst for the degradation of organic dyes

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Table S1. The experimental *d*-values and calculated *d*-values according to the indices of $H_2(H_2O)Ta_2O_6$ crystallographic planes (JCPDS Card No. 74-655).

(hkl)	experimental *d*-values (Å)	calculated *d*-values (Å)
(111)	6.0625	6.0621
(311)	3.1708	3.1659
(222)	3.0311	3.0375
(400)	2.6301	2.6250
(331)	2.4149	2.4089
(422)	2.1484	2.1433
(511)	2.0256	2.0207
(440)	1.8617	1.8562
(531)	1.7795	1.7748
(620)	1.6637	1.6602
(533)	1.6060	1.6012
(622)	1.5878	1.5829
(444)	1.5203	1.5155
(711)	1.4750	1.4703
(731)	1.3714	1.3670
(800)	1.3165	1.3125
(660)	1.2415	1.2374
(751)	1.2164	1.2124
(662)	1.2087	1.2044

In cubic system, $a = b = c$. The crystallographic lattice constants of as-obtained niobium oxide sample are calculated by using the following equation.

$$d_{hkl} = \frac{a}{\sqrt{h^2 + k^2 + l^2}} \quad (1)$$

The lattice constant a value is thus obtained by using the d-value of the sample (6.0625) and the corresponding indices of $H_2(H_2O)Ta_2O_6$ (111) crystallographic plane (JCPDS Card No. 74-655). Then other d-values of our sample can be calculated according to formula (1), which are compared with experimental d-values, as shown in Table S1.

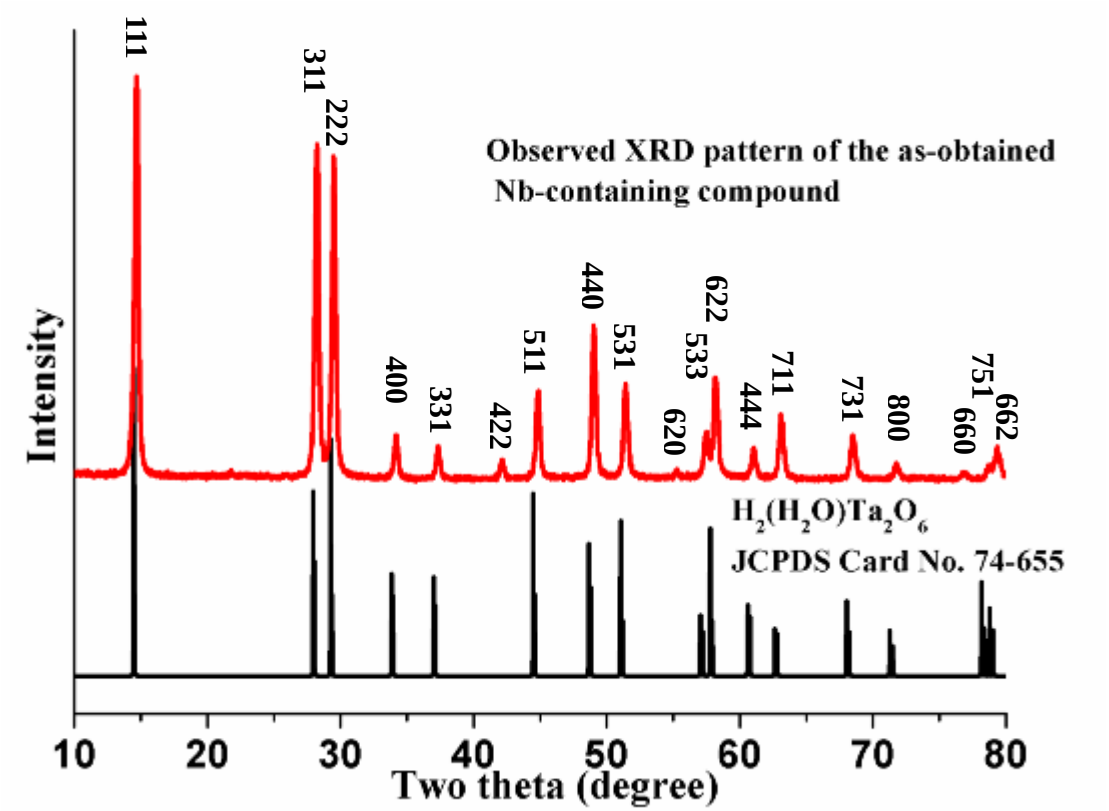

Figure S1. XRD pattern of the as-obtained Nb-containing compound. The XRD pattern is indexed according to the standard cubic $H_2(H_2O)Ta_2O_6$ pattern in JCPDS Card No. 74-655.

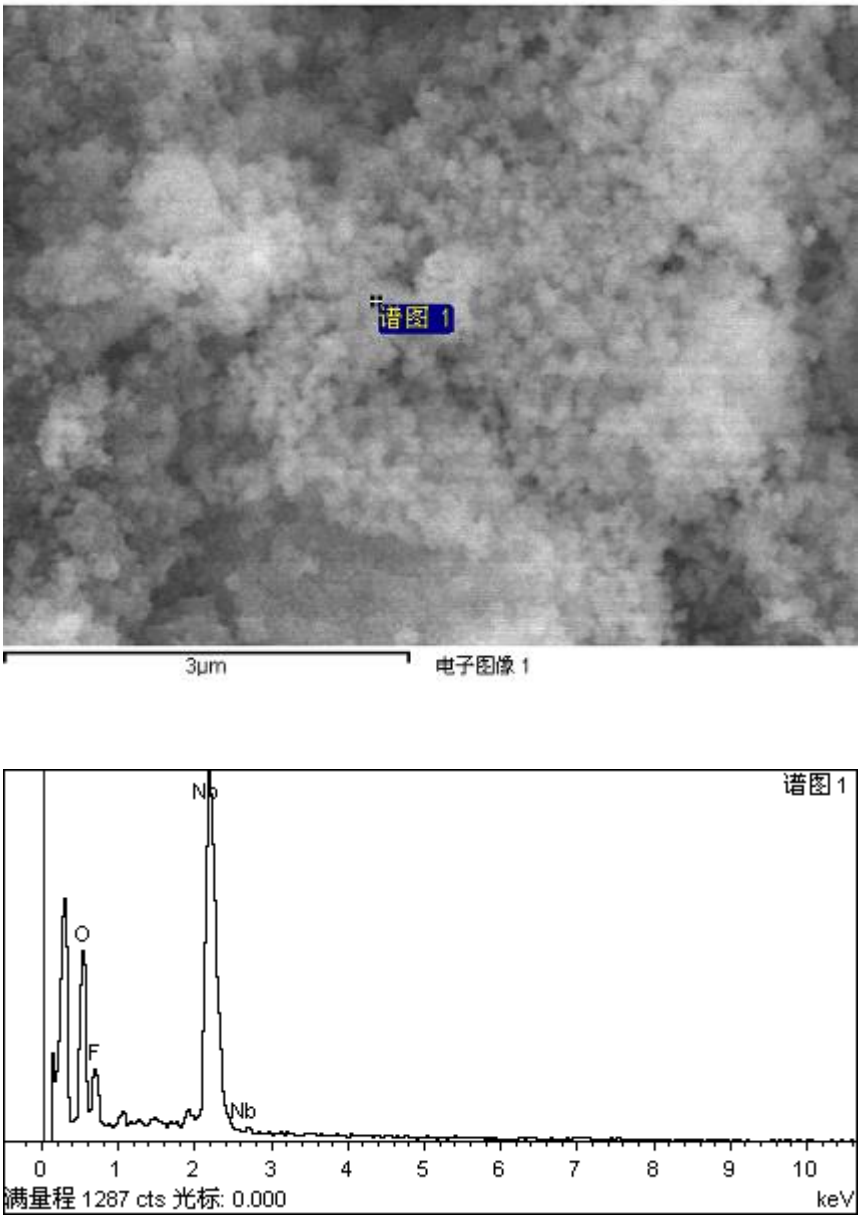

Figure S2. EDX spectrum of F-doped $H_2(H_2O)Nb_2O_6$ nanosized crystals.

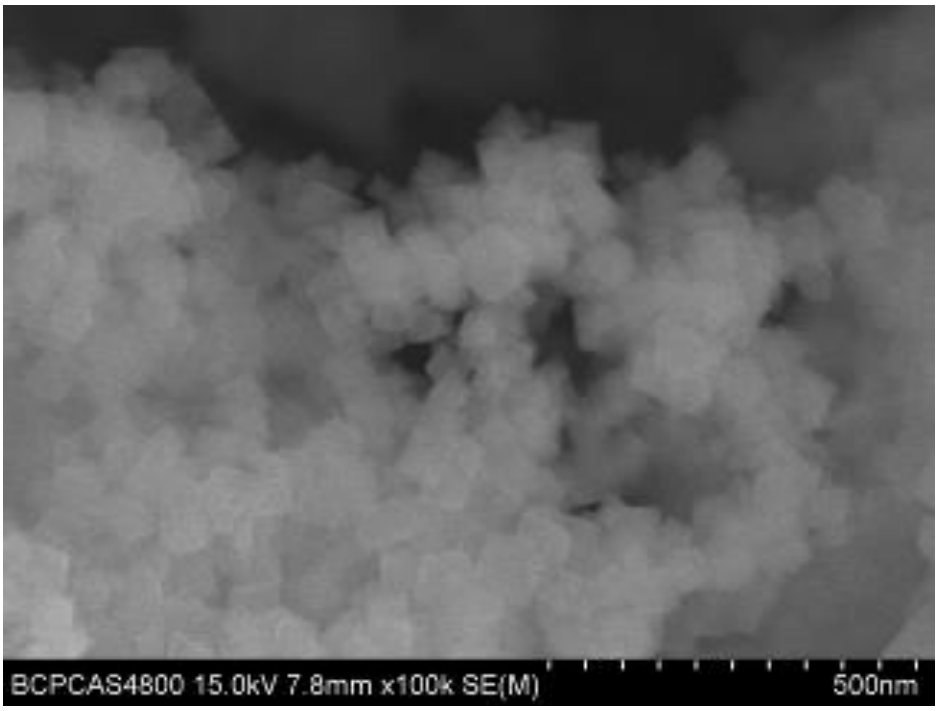

Figure S3. SEM image of the products obtained by hydrothermal treatment of amorphous $Nb_2O_5{\cdot}nH_2O$ in 10 mL aqueous solution containing NH_4F (0.20 g), $(NH_4)_2SO_4$ (0.20 g), and SDBS (0.10 g) at 160–170 °C for 120–132 h.

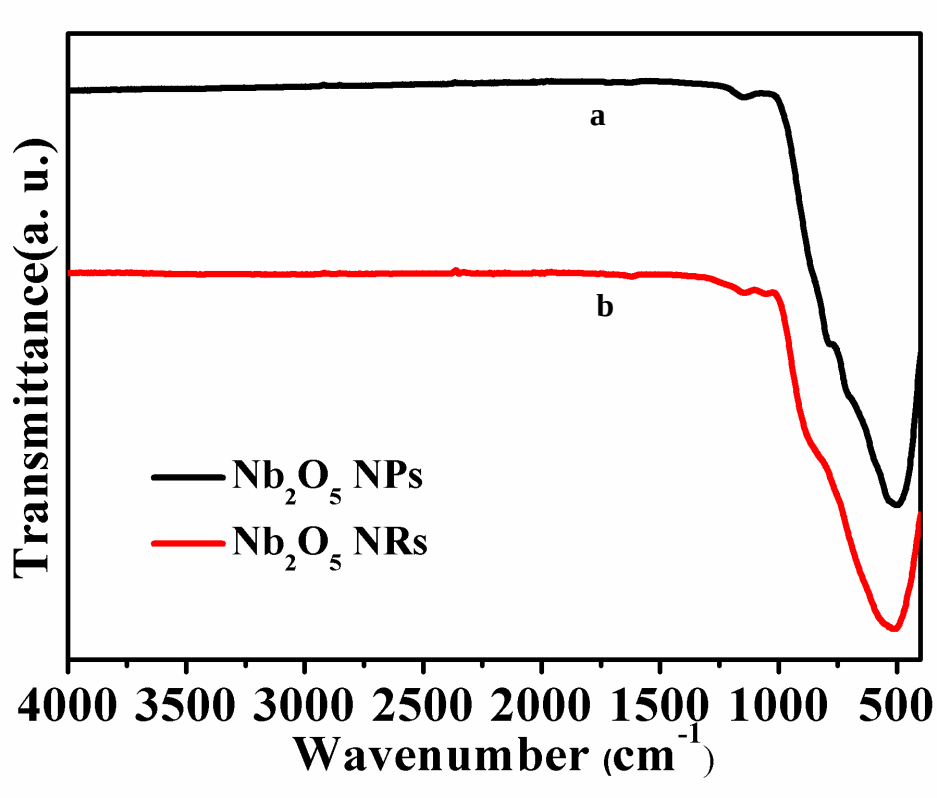

Figure S4. FT-IR spectra of Nb_2O_5 NPs obtained by calcining F-doped $H_2(H_2O)Nb_2O_6$ at 500 ^oC for 1 h and Nb_2O_5 NRs.

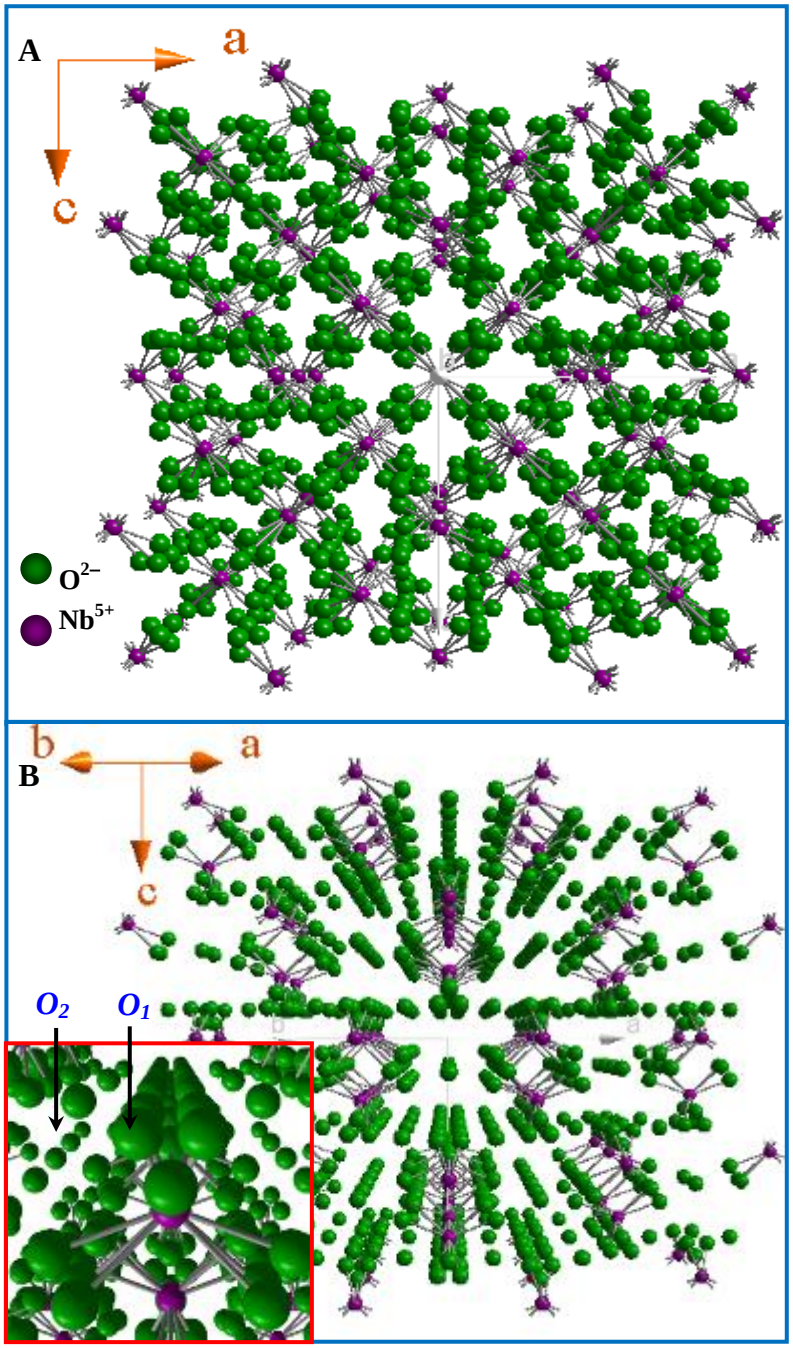

Figure S5. The crystal structure of $H_2(H_2O)Nb_2O_6$ in ball-and-stick mode, viewing along (A) *b* axis and (B) [110] direction (green: oxygen; purple: niobium). The inset in panel B shows two unique crystallographic O.

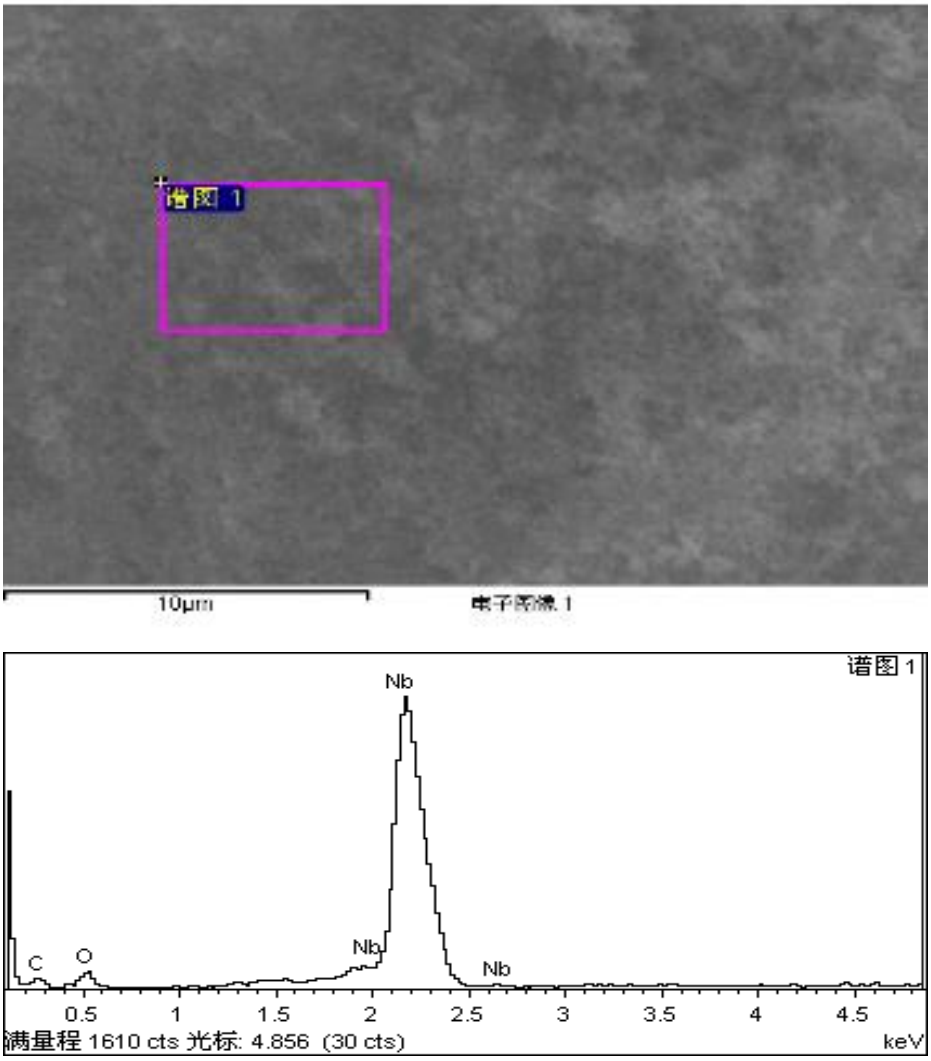

Figure S6. EDX spectrum of Nb_2O_5 NPs after calcining F-doped $H_2(H_2O)Nb_2O_6$ sample at 500 ^oC for 1 h, indicating no F element in the final product.

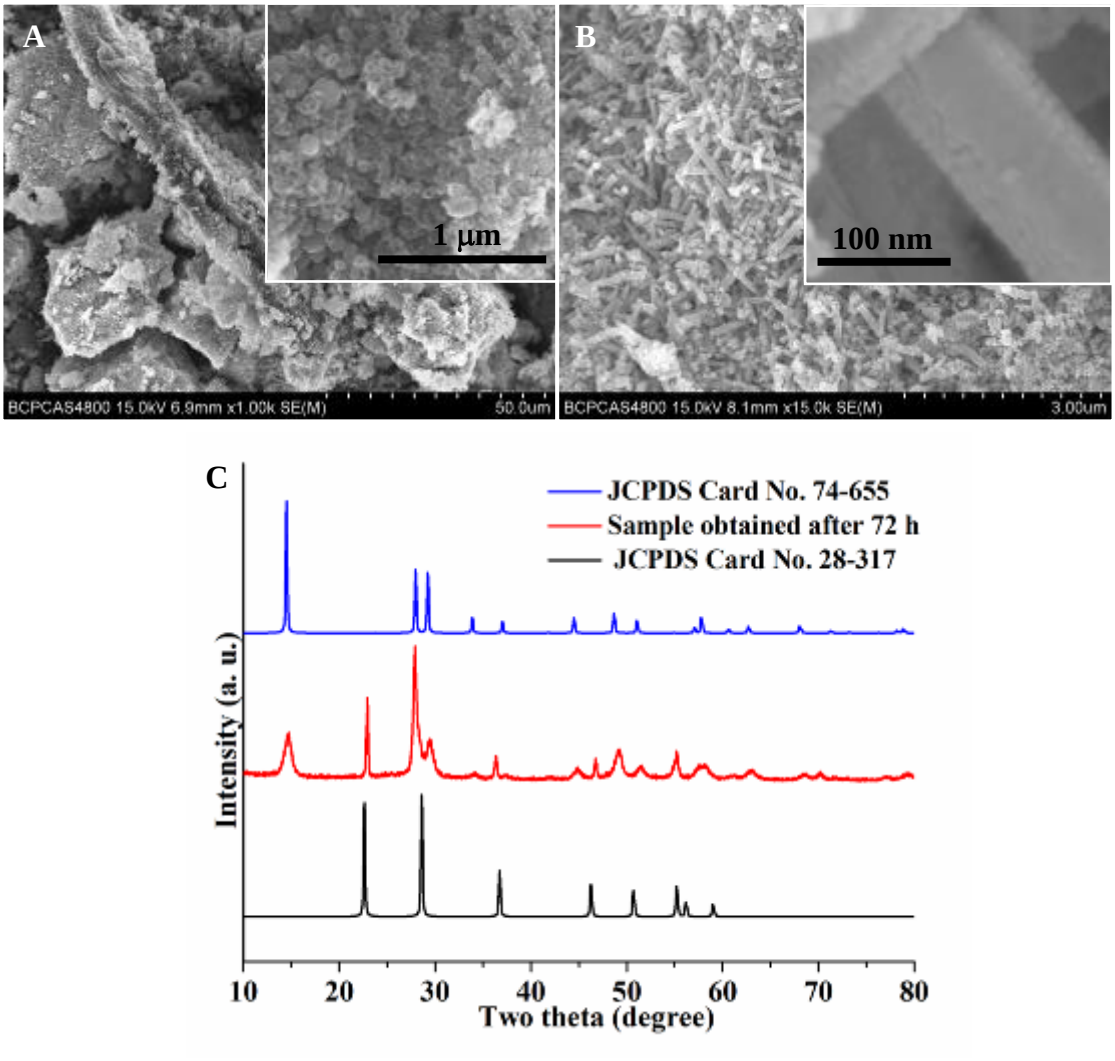

Figure S7. Typical SEM images of the products prepared at 160–170 °C with different time. (A) 14 h. (B) 72 h. (C) XRD patterns of the product obtained after 72 h reaction, showing that it is a mixture containing niobium oxide (JCPDS Card No. 28-317).

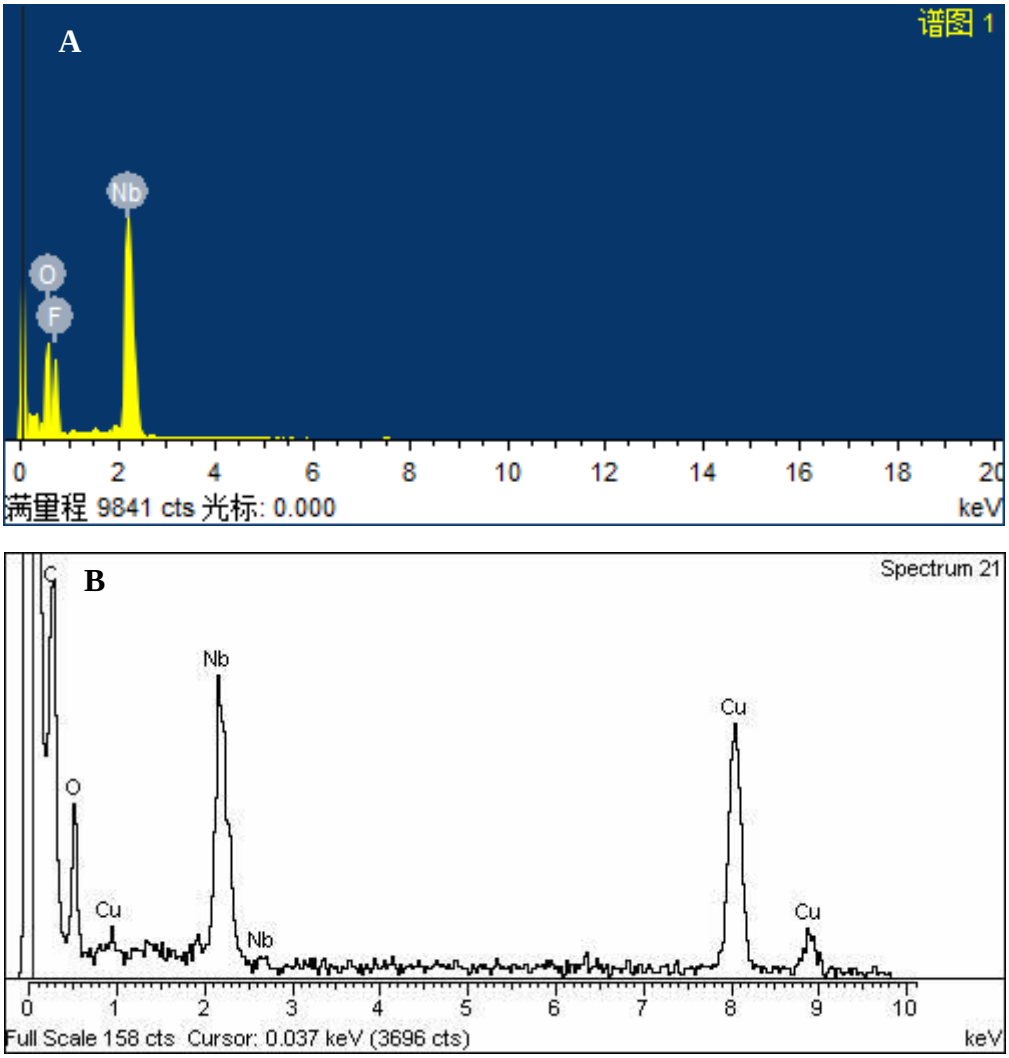

Figure S8. EDX spectra of (A) Nb_2O_5 NR-precursor and (B) Nb_2O_5 NRs obtained after calcining Nb_2O_5 NR-precursor at 700 ^{o}C for 1 h, indicating no F element in the final product (Cu signal comes from a TEM grid).

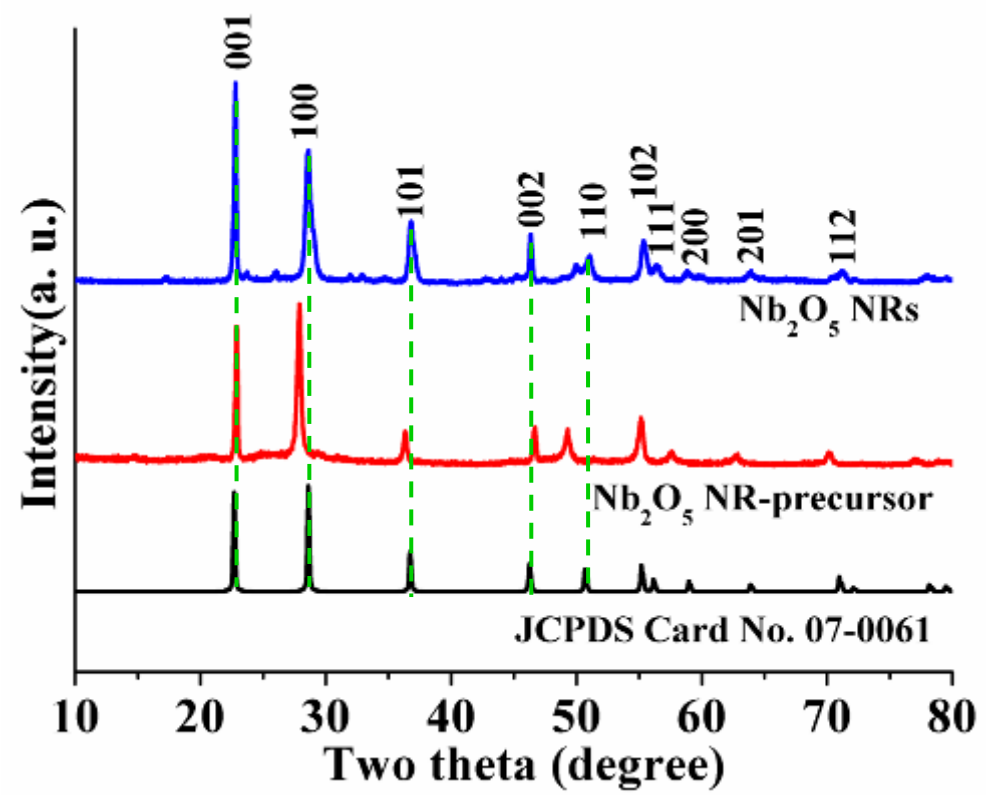

Figure S9. XRD patterns of Nb_2O_5 NR-precursor and Nb_2O_5 NRs. The XRD pattern of Nb_2O_5 NRs is indexed as the Nb_2O_5 phase (JCPDS Card No. 07-0061).

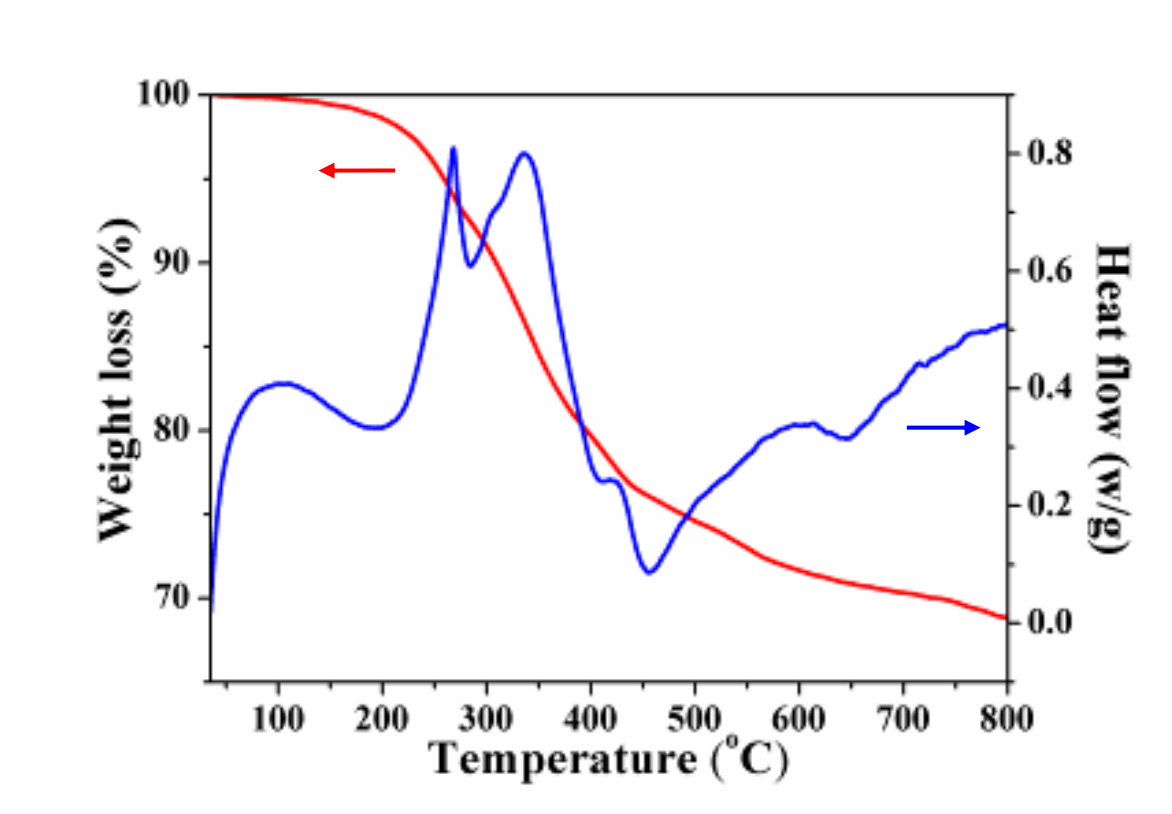

Figure S10. TG-DSC curves of Nb_2O_5 NR-precursor.

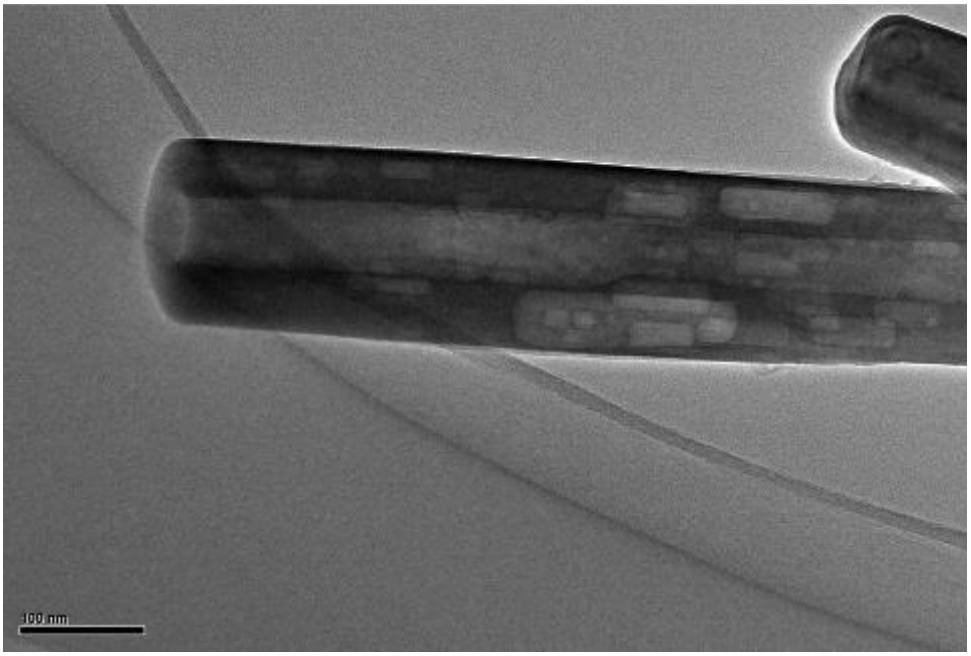

Figure S11. TEM image of Nb_2O_5 NRs, showing the porous surface after calcination at 700 ^{o}C for 1 h.

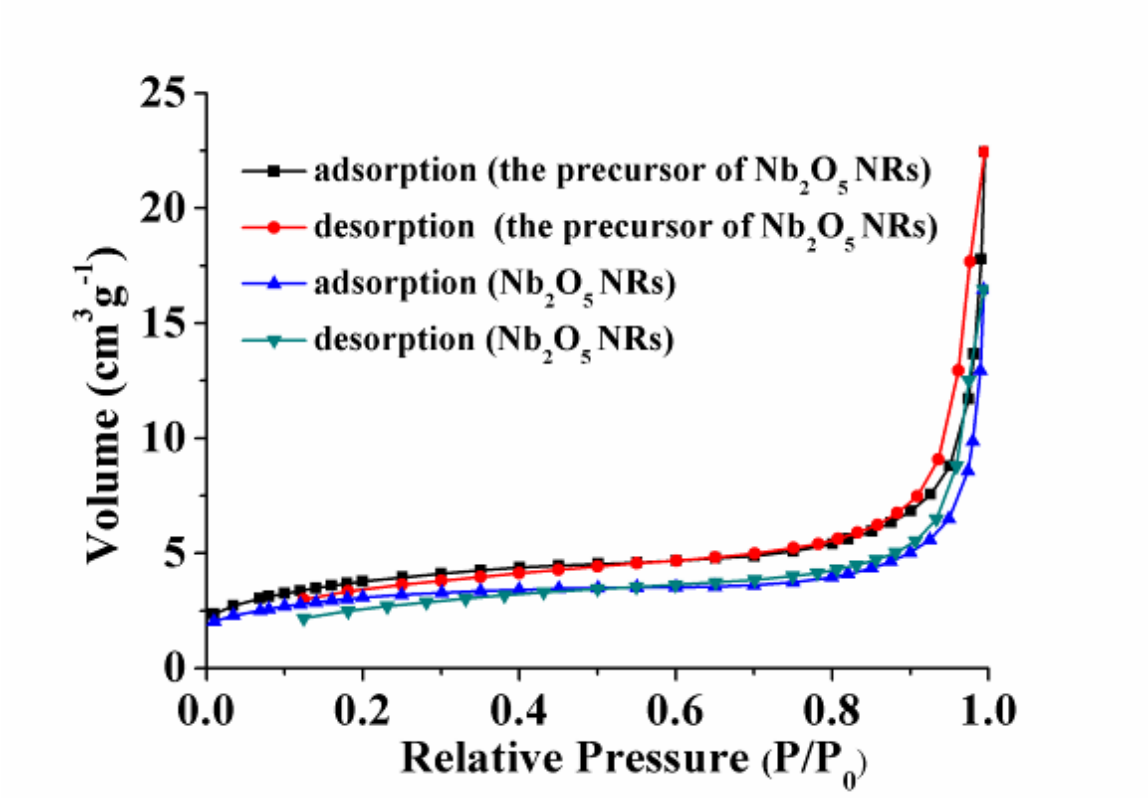

Figure S12. Nitrogen adsorption-desorption isotherms of as-prepared Nb_2O_5 NR-precursor and Nb_2O_5 NRs.

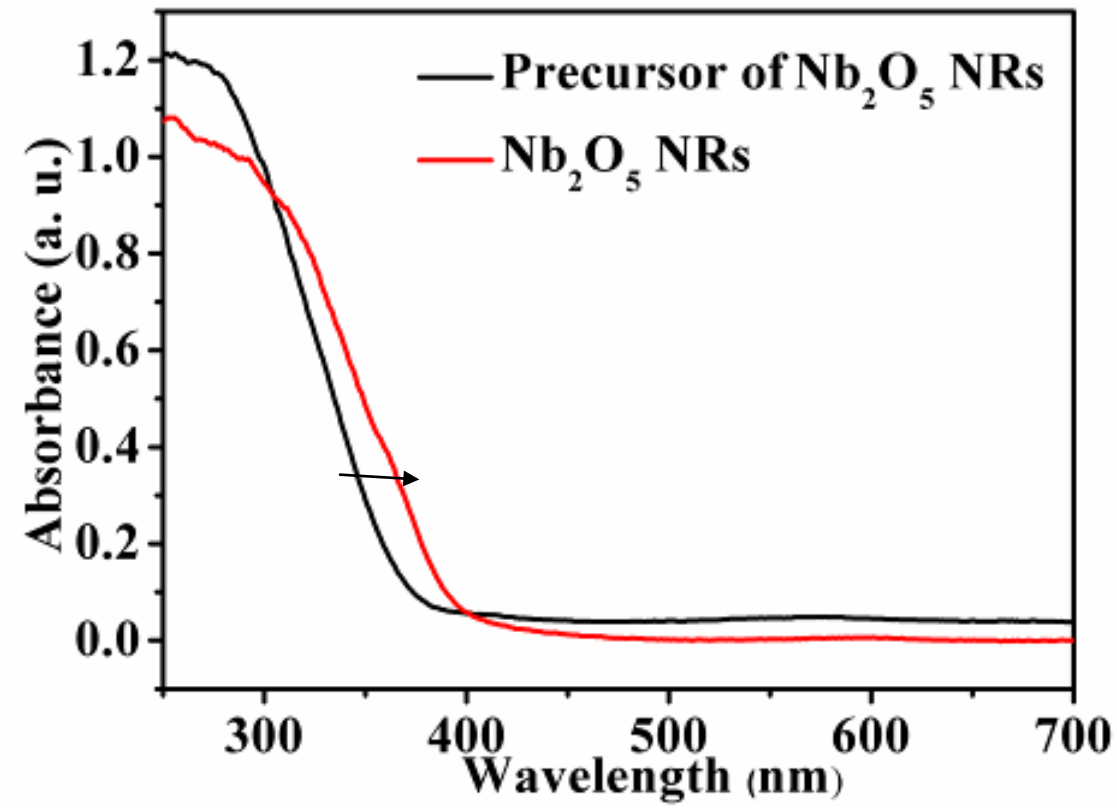

Figure S13. UV-vis absorption spectra of Nb_2O_5 NRs and Nb_2O_5 NR-precursor.

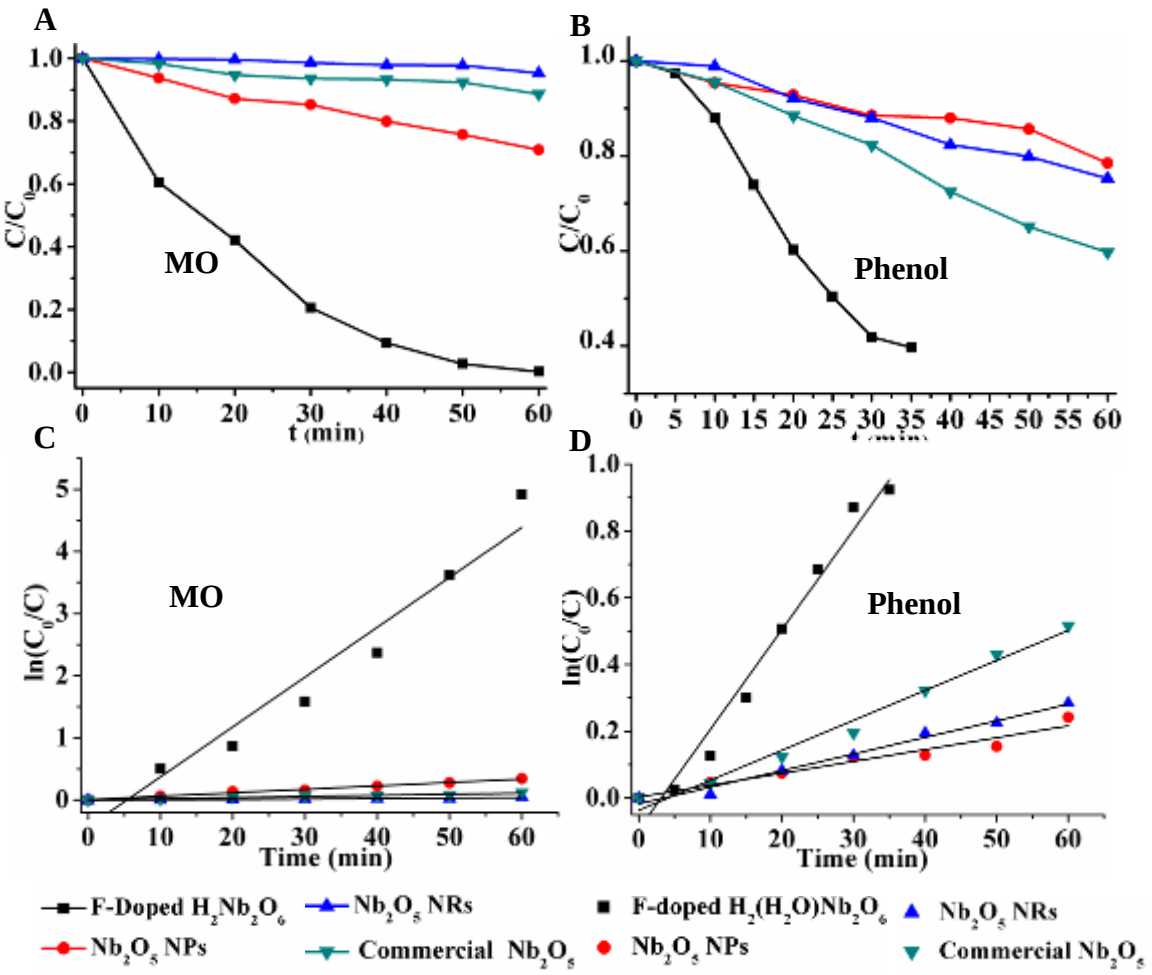

Figure S14. Photocatalytic performances of methyl orange (MO) and phenol under UV light irradiation

Table S2. The parameters of adopted pseudo first order reaction kinetics.

Samples	MB		MO		Phenol	
	k (min^{-1})	R^2	k (min^{-1})	R^2	k (min^{-1})	R^2
F-doped $H_2(H_2O)Nb_2O_6$	0.34876	0.96257	0.08025	0.94256	0.0299	0.96988
Nb_2O_5 NPs	0.12697	0.98351	0.00552	0.99002	0.00355	0.94024
Nb_2O_5 NRs	0.05106	0.96610	0.00072599	0.83176	0.00497	0.9829
Commercial Nb_2O_5 powder	0.01341	0.99114	0.0018	0.92174	0.00898	0.98049
P25-TiO_2	0.14983	0.95276				

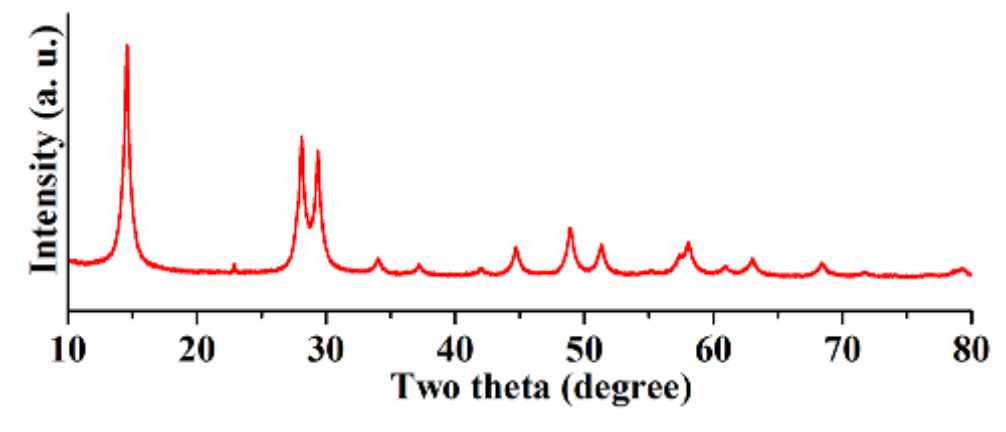

Figure S15. XRD patterns for F-doped $H_2(H_2O)Nb_2O_6$ catalysts after photodegradation of 20 mg/L MB aqueous solution for five cycles, indicating its stability when applied as a photocatalyst under UV light irradiation.