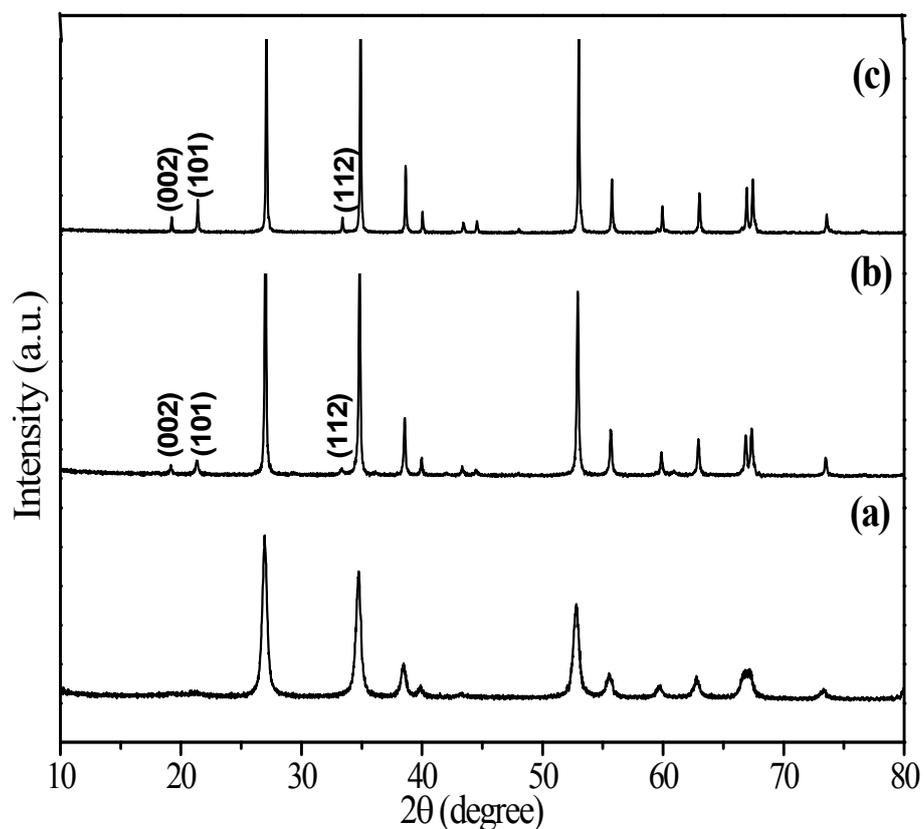


### Supplementary Materials

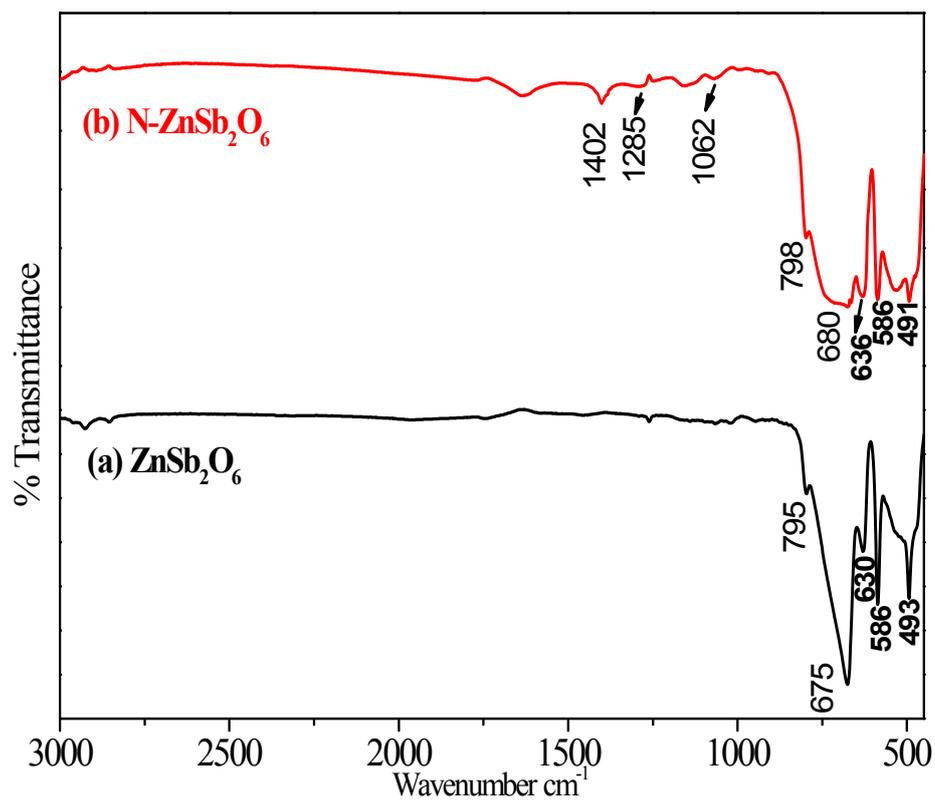
## Enhanced Photocatalytic activity of Nanocrystalline N - doped $\text{ZnSb}_2\text{O}_6$ : role of N doping, cation ordering, particle size and crystallinity

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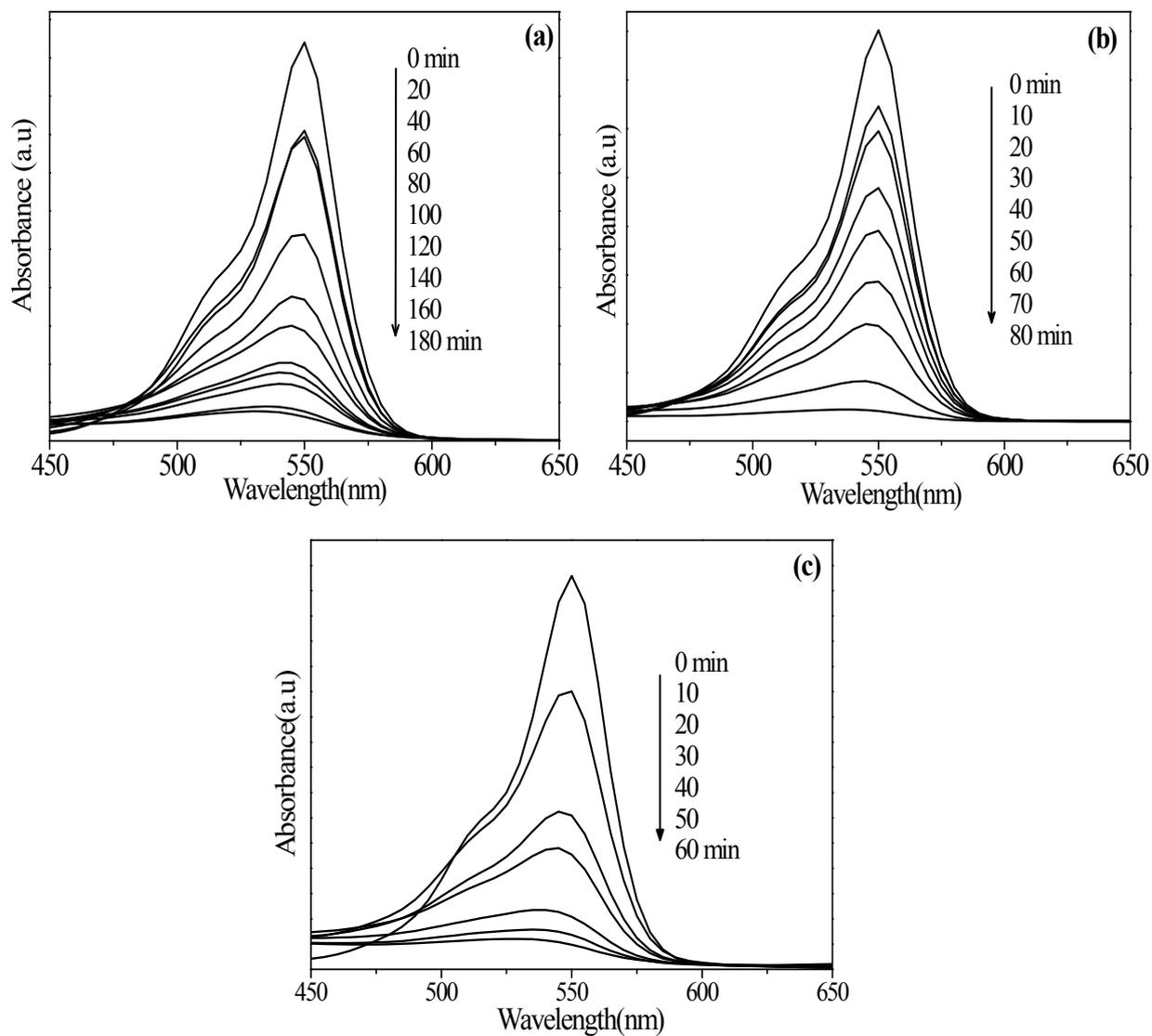
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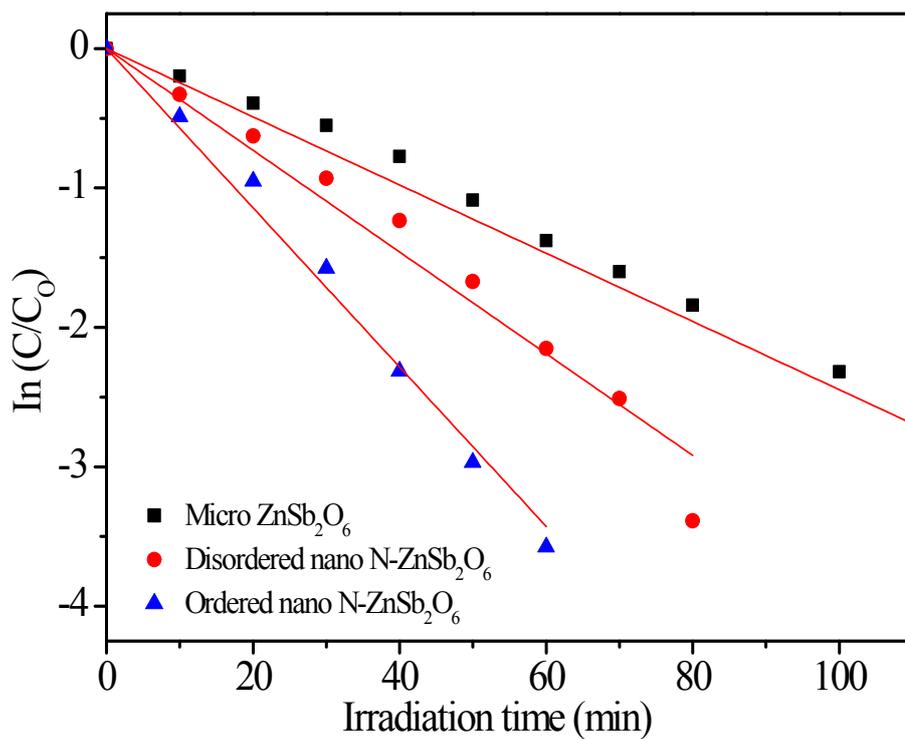
**Figure S1.** Nanocrystalline N – doped  $\text{ZnSb}_2\text{O}_6$  O/F = 1 (a) 600°C (b) 700°C and (c) 900°C/12h.



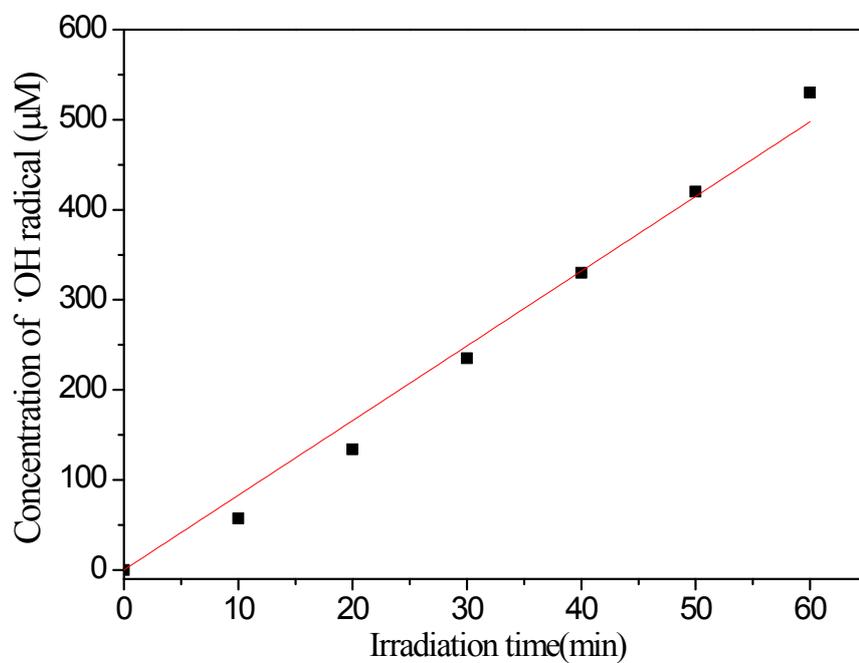
**Figure S2.** FTIR spectra of (a)  $\text{ZnSb}_2\text{O}_6$  (b) N - doped  $\text{ZnSb}_2\text{O}_6$



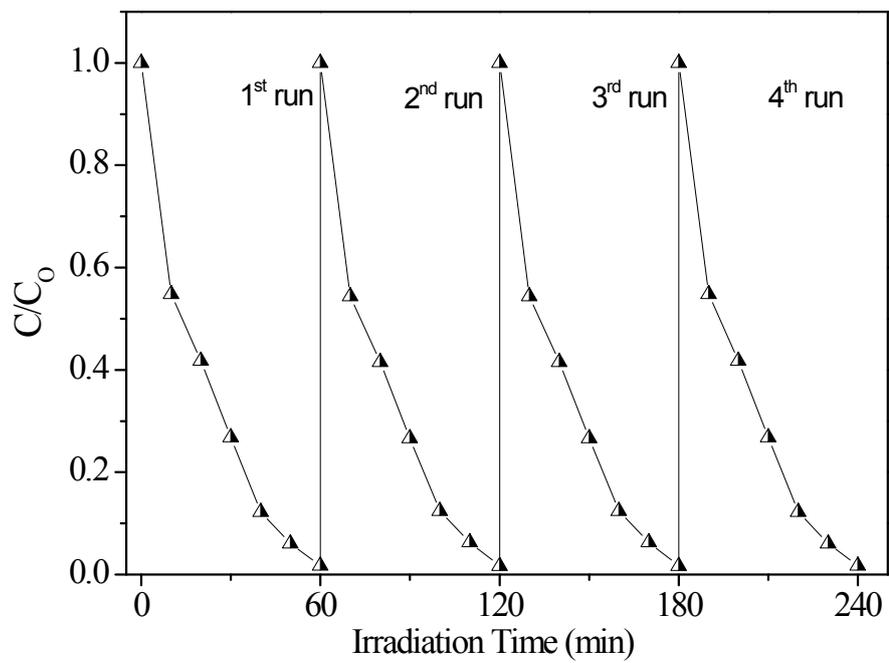
**Figure S3.** RhB photodegradation monitored by UV absorption spectra (a) ZnSb<sub>2</sub>O<sub>6</sub> (b) nanocrystalline disordered N-doped ZnSb<sub>2</sub>O<sub>6</sub> (c) ordered N-doped ZnSb<sub>2</sub>O<sub>6</sub>



**Figure S4.** Plot of  $\ln C / C_0$  with time indicating first order kinetics



**Figure S5.** Concentration of ·OH radicals from ordered N-doped ZnSb<sub>2</sub>O<sub>6</sub> with irradiation time



**Figure S6.** Recyclability of N – doped  $ZnSb_2O_6$  photocatalyst

**Table S1.** Crystallite size of nanocrystalline N - doped ZnSb<sub>2</sub>O<sub>6</sub>

O/F ratio	Crystallite size (nm)
#2	14
#1	15
#0.5	17
*600° C	26
*700° C	38
*900° C	72

# As prepared, \* O/F = 1

**Table S2.** BET surface area of microcrystalline ZnSb<sub>2</sub>O<sub>6</sub> and nanocrystalline N- doped ZnSb<sub>2</sub>O<sub>6</sub>

O/F ratio	BET Surface area m <sup>2</sup> /g
#2	34.6
#1	35.2
#0.5	31.9
*600° C	27.8
*700° C	24.7
Solid state	2.3

# As prepared, \* O/F=1

**Table S3.** Wavenumber (cm<sup>-1</sup>) of ZnSb<sub>2</sub>O<sub>6</sub> and N-doped ZnSb<sub>2</sub>O<sub>6</sub> in FTIR spectra

ZnSb <sub>2</sub> O <sub>6</sub> <sup>a</sup>	ZnSb <sub>2</sub> O <sub>6</sub> <sup>b</sup>	N-doped ZnSb <sub>2</sub> O <sub>6</sub> <sup>b</sup>	Assignment	Reference
790	795	798	Sb–O	25
700-680	675	680	O – Sb – O (SbO <sub>6</sub> )	24
640	630	636	O – Sb – O (SbO <sub>6</sub> )	24
586	586	586	Sb–O	25
491	493	491	Zn–O (ZnO <sub>6</sub> )	23
		1402	NO <sub>3</sub> <sup>-</sup> , NO <sub>2</sub> <sup>-</sup> like species	27
		1285	NH <sub>3</sub> like species	26
		1062	Zn – O – N	28
		a- literature	b- our work	

**Table S4.** Raman bands of microcrystalline ZnSb<sub>2</sub>O<sub>6</sub> and nanocrystalline N-doped ZnSb<sub>2</sub>O<sub>6</sub>

O/F	Wavenumber (cm <sup>-1</sup> )					
2	712	633	515	305	275	
1	722	646	519	306	279	
0.5	725	650	521	311	280	
Microcrystalline	736	657	528	319	283	

**Table S5.** Photocatalytic activity of microcrystalline ZnSb<sub>2</sub>O<sub>6</sub> and nanocrystalline N- doped ZnSb<sub>2</sub>O<sub>6</sub>

Compound	Degradation %	Complete degradation time (min)	k x10 <sup>-2</sup> min <sup>-1</sup>	•OH radical concentration (μM)
Ordered N-ZnSb <sub>2</sub> O <sub>6</sub>	99	60	6.34	8.5
Disordered N-ZnSb <sub>2</sub> O <sub>6</sub>	99	80	4.93	6.8
ZnSb <sub>2</sub> O <sub>6</sub>	96	180	1.72	2.9