Electronic Supplementary Material (ESI) for CrystEngComm

One-Step Template-free Synthesis of BaSb₂O₆ Micro-flowers and Their Associated Photocatalytic Activity

Jing Chen, Danzhen Li^{*}, Junhua Hu, Wei Chen, Jinxiu Wang, Yin Hu, Xianzhi Fu, Yu Shao

Research Institute of Photocatalysis, State Key Laboratory Breeding Base of Photocatalysis, Fuzhou University, Fuzhou, 350002, P. R. China Tel & Fax: (+86)591-83779256; E-mail: <u>dzli@fzu.edu.cn</u>



Figure S1. XRD patterns of the R-BaSb₂O₆ formed during different reaction time



Figure S2. XRD patterns of the R-BaSb₂O₆ synthesized at different temperatures (a), variation of R-BaSb₂O₆ morphologies with reaction temperature from 120 to 200 $^{\circ}$ C

$$(b) \sim (d).$$

The reaction temperature related variation of R-BaSb₂O₆ morphologies is investigated. As shown in Figure S2, the rose-flower-like 3D hierarchical structures can be obtained at the range of 120~200 °C reacting for 48 hr at pH=3. However, only the samples synthesized at 200 °C shows the clear edges and sharp angles. While the samples synthesized at 160 °C and 120 °C are round and their edges are fuzzy. So, all the other samples in this paper were synthesized at 200 °C.

| pH values | 1 | 2 | 3 | 4 | 5 |
|------------------------------------|----|-----|-----|-----|---|
| Scale of the | | | | | |
| hexagonal plate | | | | | |
| or the | 16 | 11 | 7 | 8 | 7 |
| hierarchical | | | | | |
| structures ¹ (μ m) | | | | | |
| Thickness of one | 1 | 0.7 | 0.1 | 0.1 | |
| layer (µm) | 1 | 0.7 | 0.1 | 0.1 | |

Table S1 The scales of samples synthesized at different pH values of precursor

solution at 200 °C for 48 hr

1 Length of the longest diagonal of the hexagonal plate or the diameter of the hierarchical structures (μm).



Figure S3. XRD pattern of the white precipitate at 200 °C with pH=0.5 for 48 hr.



Figure S4. XRD patterns of M-BaSb₂O₆ formed during different reaction time.



Figure S5. XRD pattern of SSR-BaSb₂O₆



Figure S6 XPS of SSR-BaSb $_2O_6$ and flower-like BaSb $_2O_6$



Figure S7 DRS of flower-like $BaSb_2O_6$ and $SSR-BaSb_2O_6$