Electronic Supplementary Material (ESI) for Journal of Materials Chemistry C.

Supporting Information

Synthesis of $Bi_{2-2x}Sb_{2x}S_3$ ($0 \leq x \leq 1$) Solid Solutions from Solventless Thermolysis of Metal Xanthate Precursors

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Table S1: Composition of $Bi_{2-2x}Sb_{2x}S_3$ ($0 \leq x \leq 1$).

Composition (*x*) [Sb]/[Sb]+[Bi]	**$Bi[S_2COEt]_3$ (1)**	**$Sb[S_2COEt]_3$ (2)**
0	0.35 mmol	0
0.2	1.64 mmol	0.41 mmol
0.4	0.61 mmol	0.41 mmol
0.6	0.21 mmol	0.41 mmol
0.8	0.11 mmol	0.41 mmol
1	0	0.41 mmol

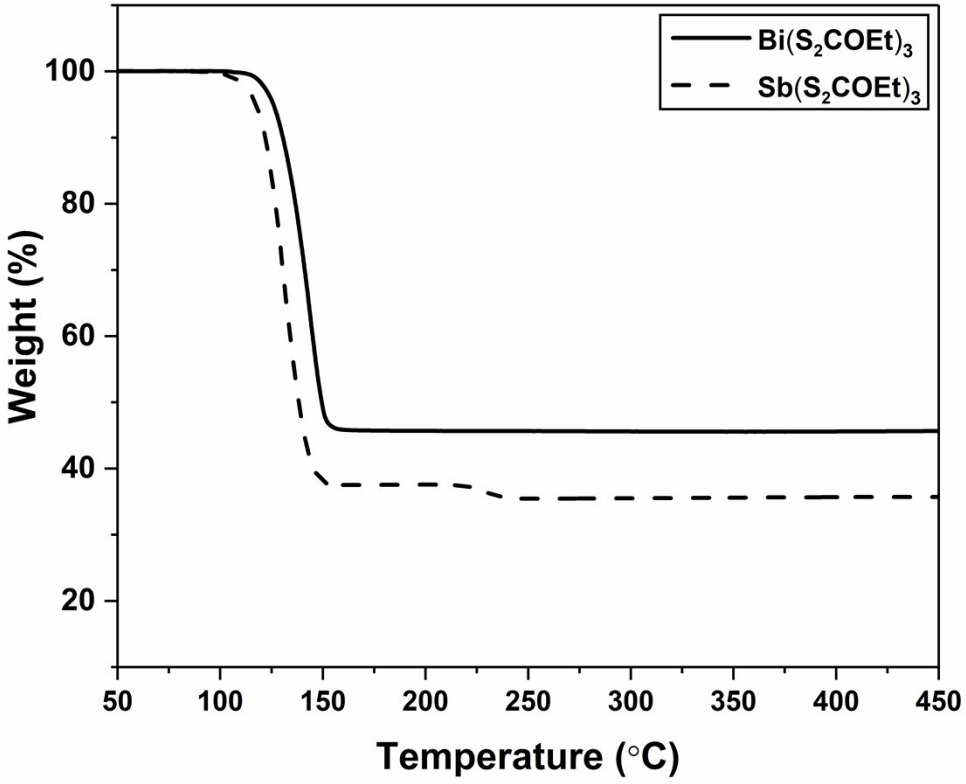

Figure S1: TGA for bismuth(III) and antimony(III) ethylxanthate.

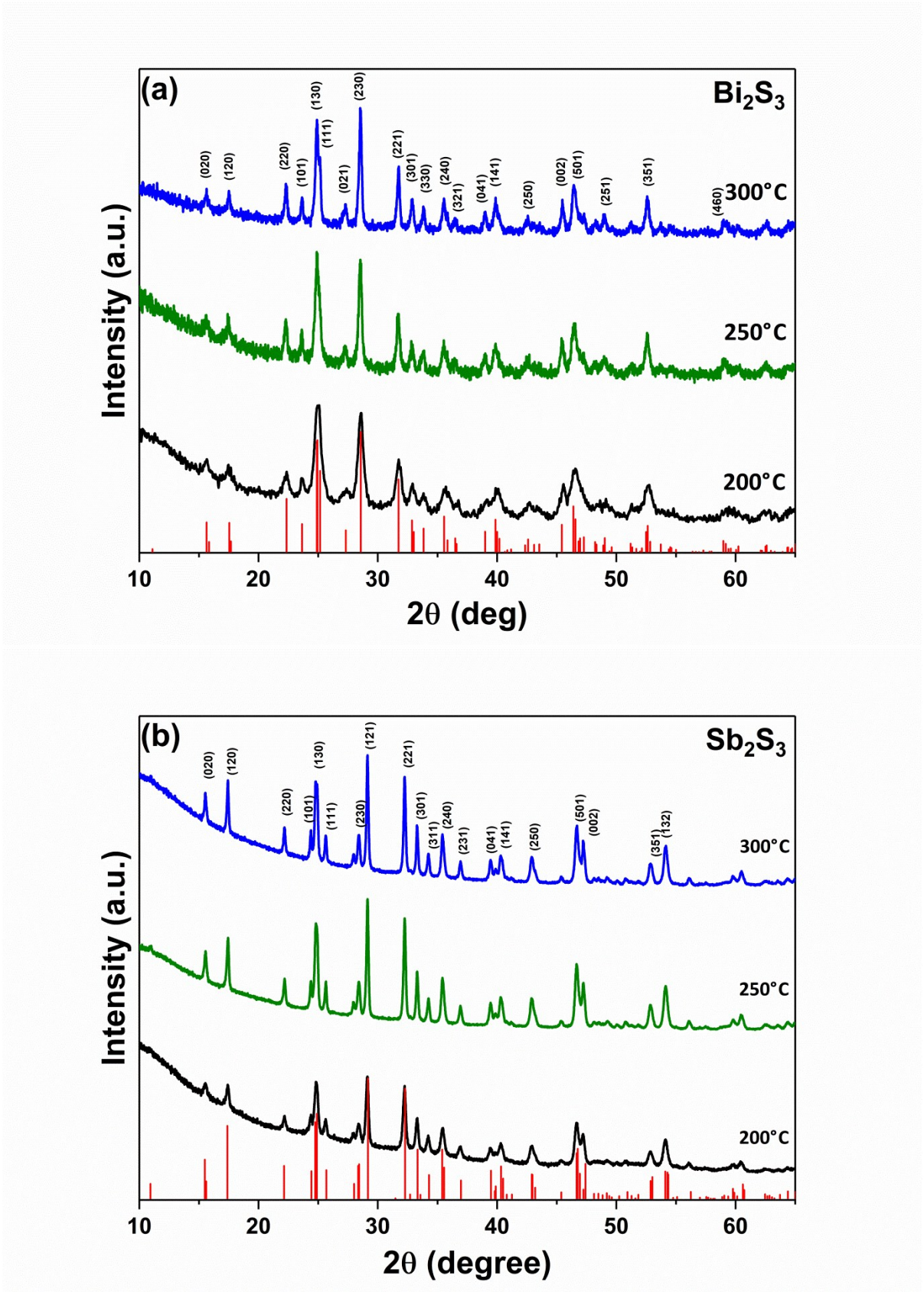

Figure S2: XRD for (a) Bi_2S_3 from bismuth(III) ethylxanthate and (b) Sb_2S_3 from antimony(III) ethylxanthate at different temperatures.

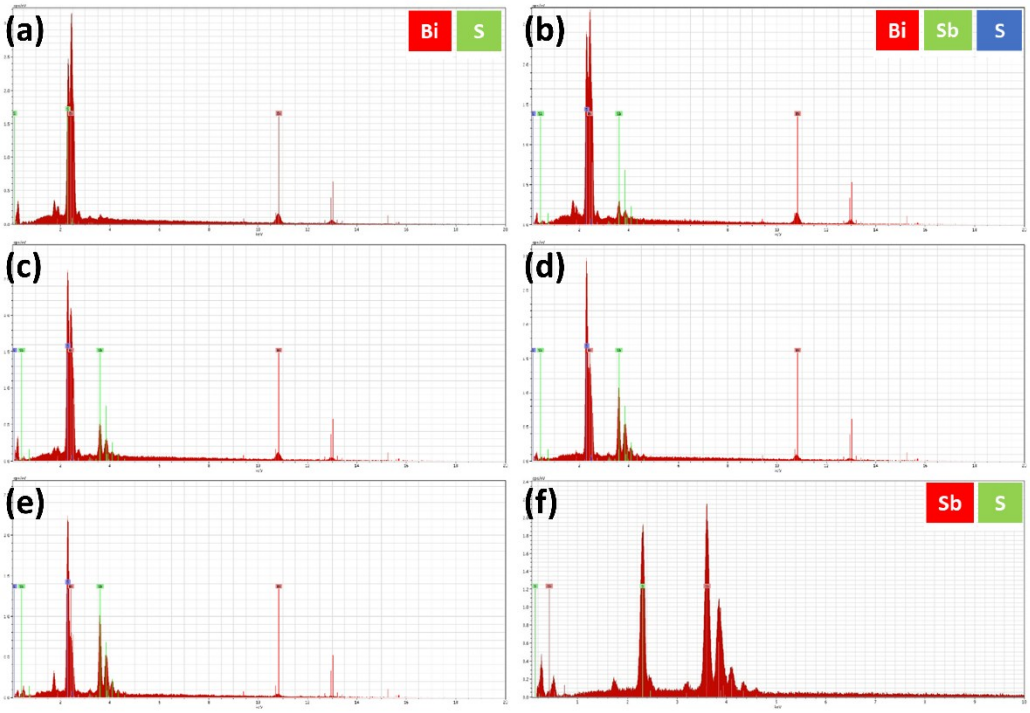

Figure S3: EDX spectra of $Bi_{2-2x}Sb_{2x}S_3$ with (a) x = 0, (b) x= 0.2, (c) x = 0.4, (d) x = 0.6, (e)x = 0.8 and (f) x=1.

Table S2: The content of Bi, Sb and S in $Bi_{2-2x}Sb_{2x}S_3$ ($0 \leq x \leq 1$) calculated from theoretical values, EDX and ICP-OES.

Sb mole fraction	Element	Atomic % (Required composition)	Atomic% (Composition by EDX)	Atomic% (Composition by ICP-OES)
0	**Bi** **Sb** **S**	**40** **0** **60**	**43.6** **0** **56.4**	**42.5** **0** **57.5**
0.2	**Bi** **Sb** **S**	**32** 8 60	**36.9** **10.8** **52.3**	**32.8** **11.1** **56.1**
0.4	**Bi** **Sb** **S**	**24** 16 60	**27.6** **17.7** **54.6**	**27.1** **20.3** **52.6**
0.6	**Bi** **Sb** **S**	**16** 24 60	**17.3** **27** **55.6**	**13.7** **28.2** **58.1**
0.8	**Bi** **Sb** **S**	**8** 32 60	**8.4** **33** **58.5**	**9.2** **34.3** **56.5**
1	**Bi** **Sb** **S**	**0** **40** **60**	**0** **40.6** **59.4**	**0** **45.5** **54.5**

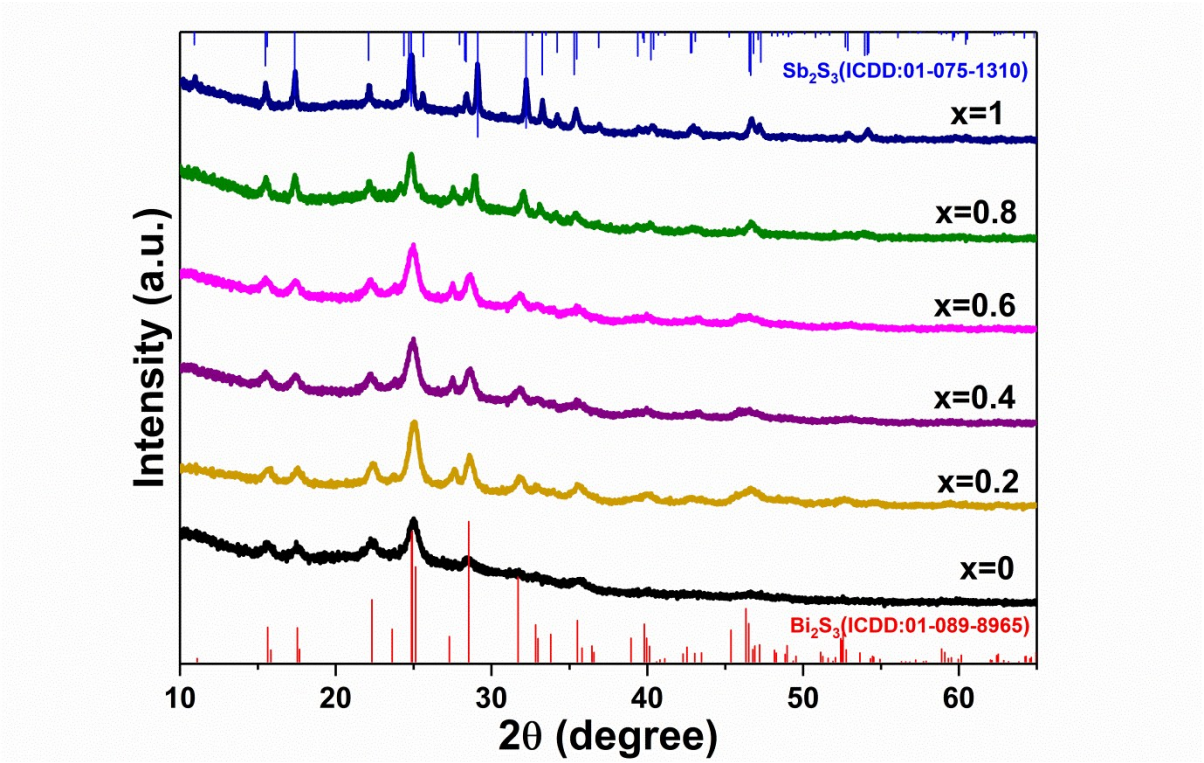

Figure S4: XRD for $Bi_{2-2x}Sb_{2x}S_3$ ($0 \leq x \leq 1$) films prepared by spin coating method at 250 °C.

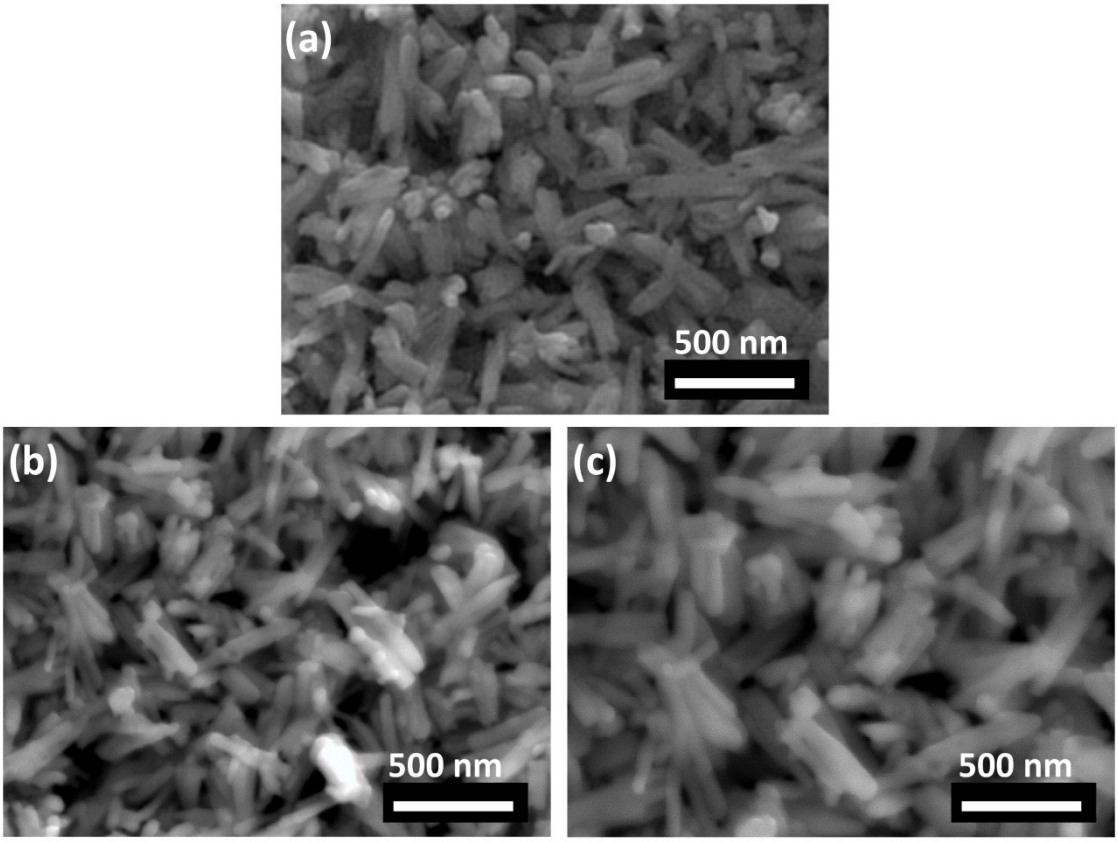

Figure S5: SEM images at low magnification showing the nanorods structure of $Bi_{2-2x}Sb_{2x}S_3$ at 250 °C using different mole fractions of Sb (a) Bi_2S_3 (x = 0), (b) 0.2 and (c) 0.4.

Table S3: Lattice spacings, $\mathbf{d_{ED}}$*, measured using electron diffraction (ED) data (with associated uncertainties*). ED *data presented in main text Figure 8. ED data is compared to expected lattice spacings,* $\boldsymbol{d_{p\text{-}XRD}}$*, predicted from* 2θ values determined by *p-XRD.*

x = 0.2			
(hkl)	**2θ (°)**	**$d_{p\text{-}XRD}$ (Å)**	**d_{ED} (Å)**
020	15.64	5.66	5.71 ± 0.59
120	17.53	5.06	5.00 ± 0.55
220	22.32	3.98	3.99 ± 0.51
130	24.88	3.58	3.62 ± 0.47
121			3.11 ± 0.44
221	31.77	2.81	2.79 ± 0.42
301	32.87	2.72	2.68 ± 0.42
240	35.51	2.53	2.49 ± 0.43
140	39.85	2.26	2.25 ± 0.41
241			2.11 ± 0.41
501	46.54	1.95	1.94 ± 0.37
251			1.84 ± 0.40
351	52.56	1.74	1.71 ± 0.38
242			1.55 ± 0.39
342			1.48 ± 0.39
461			1.44 ± 0.39
800			1.42 ± 0.39

x = 0.8			
(hkl)	**2θ (°)**	**$d_{p\text{-}XRD}$ (Å)**	**d_{ED} (Å)**
020	15.62	5.67	5.66 ± 0.57
120	17.51	5.06	5.04 ± 0.54
220	22.26	3.99	3.90 ± 0.50
130	24.85	3.58	3.57 ± 0.47
111			3.42 ± 0.49
211			3.05 ± 0.46
221	32.25	2.77	2.75 ± 0.43
301	33.30	2.69	2.67 ± 0.42
240	35.46	2.53	2.55 ± 0.42
231			2.45 ± 0.41
321			2.40 ± 0.41
140	40.32	2.23	2.23 ± 0.39
421			2.10 ± 0.39
501	46.72	1.94	1.96 ± 0.38
450			1.75 ± 0.36
351	52.76	1.73	1.73 ± 0.36

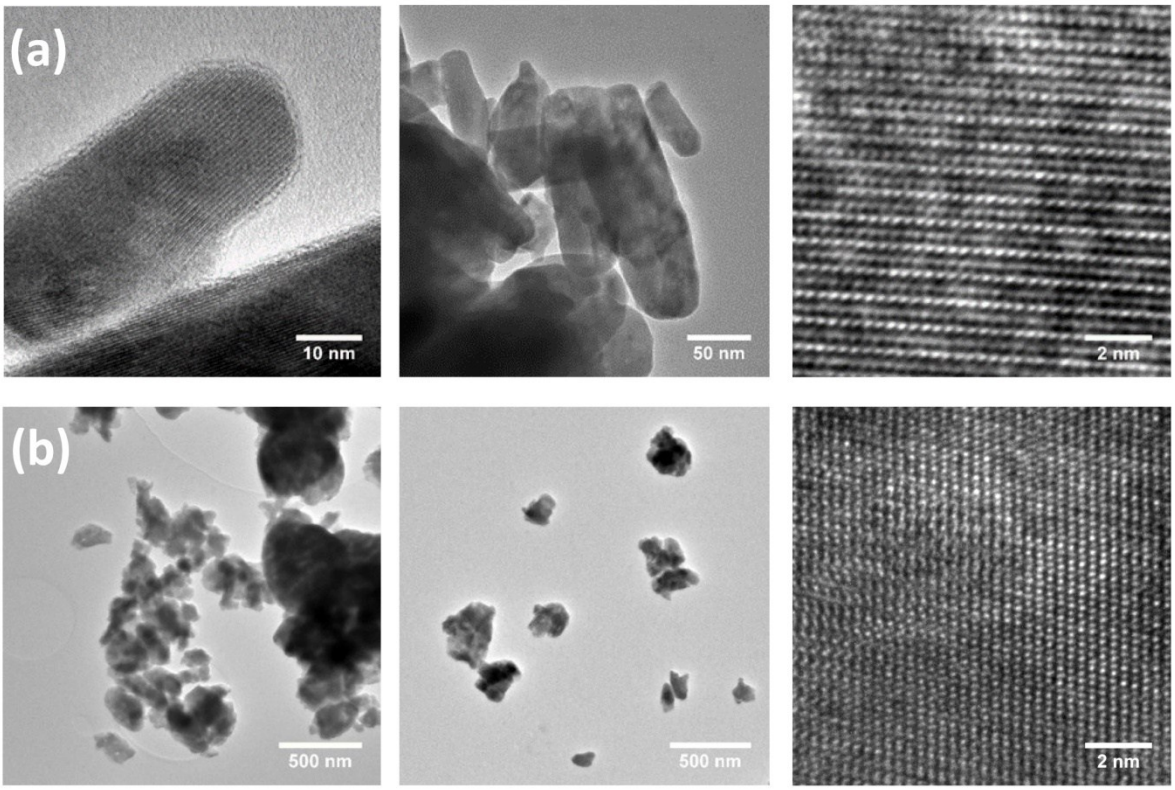

Figure S6: TEM images of $Bi_{2-2x}Sb_{2x}S_3$ (a) x = 0.2 and (b) x = 0.8.

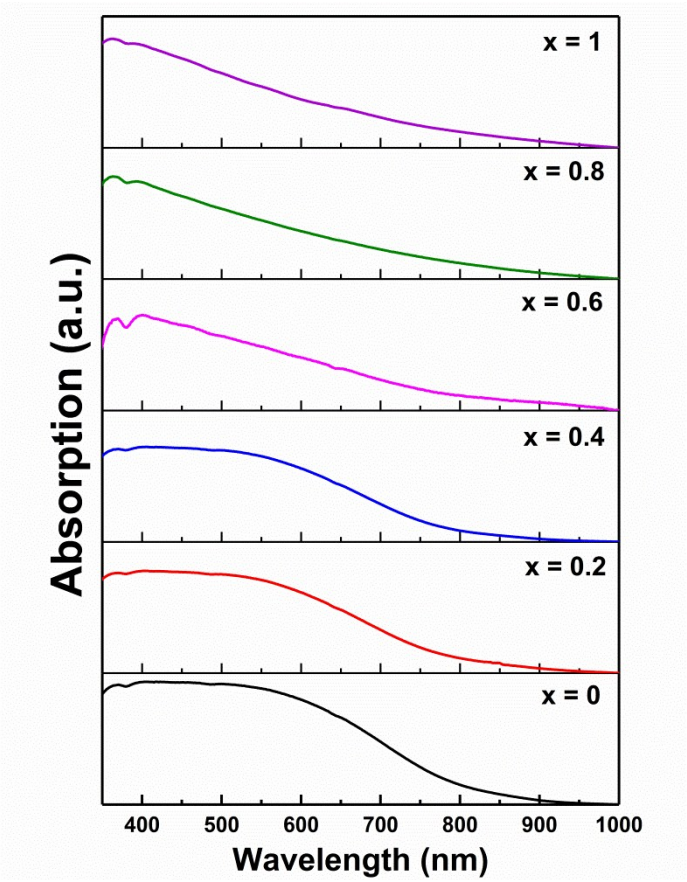

Figure S7: Absorption spectra of $Bi_{2-2x}Sb_{2x}S_3$ ($0 \leq x \leq 1$).

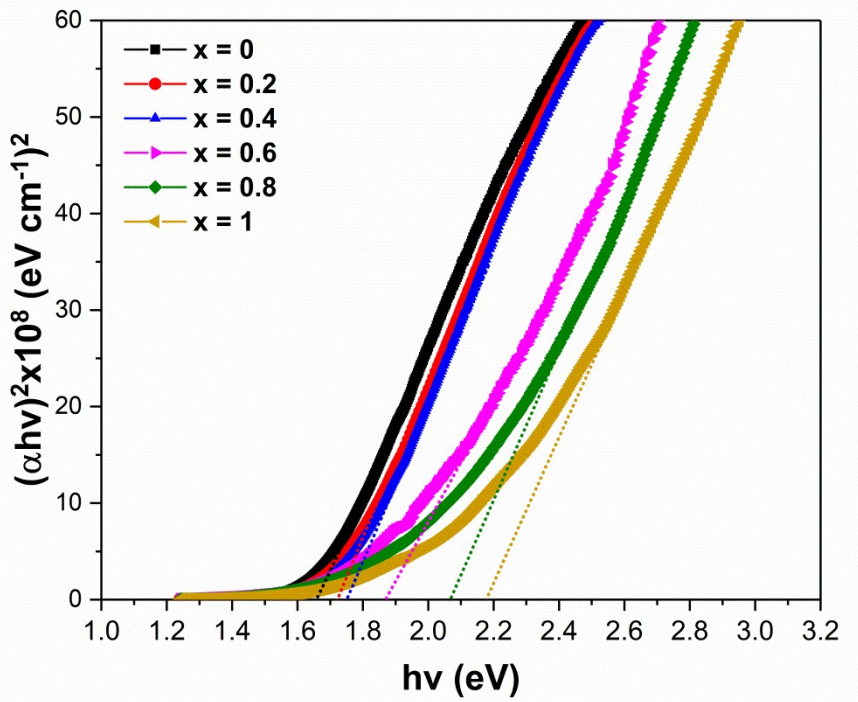

Figure S8: Tauc plot for the $Bi_{2-2x}Sb_{2x}S_3$ with different x values: (a) x = 0 and 1, (b) x = 0.2, 0.4, 0.6 and 0.8.