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Solventless synthesis of antimony sulfide, bismuth sulfide, and antimony-bismuth sulfide solid solutions using novel single source route

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Antimony(III) ethylxanthate [$Sb(S_2COEt)_3$] and bismuth(III) ethylxanthate [Bi(S2COEt)3] were used as a single source precursors for the preparation of Sb_2S_3 and Bi_2S_3, respectively, by a melt method at different temperatures. In addition, the thermogravimetric analysis reveals that both precursors exhibit complete decomposition in similar temperature range. Therefore, the mixture of these precursors can be used to produce solid solutions of Bi-Sb-S between the two phases (Bi_2S_3 and Sb_2S_3). A series with varying stoichiometry was synthesized by using different molar ratios (i.e. Sb/Sb+Bi=0.2, 0.4, 0.6 and 0.8). The XRD peaks at all ratios correspond well to the orthorhombic crystals, where the peaks fall in between those of orthorhombic Bi_2S_3 and orthorhombic Sb_2S_3 for Bi-Sb-S system. The gradual splitting and shift in the peaks position confirms the successful incorporation of antimony into bismuth sulfide. The inclusion of antimony was further confirmed by change in lattice parameters and is in good agreement with the literature values. A decrease of almost 3.5% in volume was observed as moving from Bi_2S_3 to Sb_2S_3. A change in all lattice parameters indicates that the substitution is random and not in any specific direction. The elemental compositions of all the samples were examined via energy-dispersive X-ray spectroscopy (EDX) analysis and Inductively coupled plasma–optical emission spectrometry (ICP-OES), which shows uniform distribution of elements in all samples. The morphology for all the samples was observed using SEM, revealing different morphologies as the composition changes from Bi_2S_3 to Sb_2S_3.

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