International Journal of Education and Science Research Review
E-ISSN 2348-6457
Volume-10, Issue-1 Jan-Feb-2023
P-ISSN 2349-1817
www.ijesrr.org
Email- editor@ijesrr.org

Topic: Synthesis of bimetallic $FeSb_2$nanoparticles through wet chemical approach

1. Suman Lata Pandey*
Research Scholar
Suman.pandey108@gmail.com
Department of Pharmacy, Sunrise University, Alwar, Rajasthan

2. Dr. Abhishek Kumar*
Research Guide
Department Of Pharmacy, Sunrise University, Alwar, Rajasthan

Intermetallic nanocrystals show promising magnetic, thermoelectric, and electronic properties. Nonetheless, nanorange synthesis methods are scarce, which restricts their exploration in applications. Here, we report a powerful and economical one-pot polyol approach for the synthesis of low-temperature thermoelectric FeSb2 nanoparticles. The structure of FeSb2 was confirmed by X-ray diffraction (XRD). The formation of spherical particles in the range of 10-20 nm was confirmed by scanning electron microscope (SEM) and transmission electron microscope (TEM). Furthermore, the thermal stability and magnetic nature of particles were analysed by thermogravimetric analysis (TGA) and superconducting quantum interference device (SQUID) magnetometer, respectively.

Introduction

Intermetallics, consisting of transition-metal and main group elements, gain tremendous attraction in the research community, due to their multifunctionality in various fields such as hydrogen storage, thermoelectric, spintronics, shape memory effect, superconductivity, etc. [1-2]. The unique character of adopting specific crystal and electronic structure compared toits constituent elements, makes them structurally and mechanically stable [3].

Intermetallic $FeSb_2$ has been well explored in the field of thermoelectric for cryogenic applications as it exhibits a huge Seeback coefficient of 45000 $\mu V\ K^{-1}$ at temperatures around 10 K [4-5]. But the very high value of its thermal conductivity limits the overall thermoelectricperformance and hence restricts its use for practical applications [6]. Several studies revealed an improvement in the thermoelectric performance of materials by introducing more scatteringpoints in the system, i.e., nano structuring of the materials [7].

Synthesis of intermetallic compounds generally requires a high reaction temperature, which is possible in physical techniques such as arc-melter, sputtering, flux growth, chemical vapor transport, etc. [1] But, the main disadvantage of these routes is bulk sampling as it again requirestop-down approaches such as ball-milling for the nano structuring of the material. Therefore, the direct synthesis of nanosized materials using a bottom-up approach is the need of the hour.wet-chemical synthesis of $FeSb_2$ has previously been reported in literature via solvothermal [8-10], low-temperature molten-

salt [11], and modified polyol approaches [1, 12].

In this contribution, we report the successful synthesis of $FeSb_2$ nanoparticles via a simple andeconomical one-pot polyol (high-boiling solvent) approach without adding any external surfactant or reducing agent. It is worth highlighting the main advantage of this method to solvent (polyol) reusability, which makes it a green synthesis method.

Result and Discussion

X-ray analysis: The XRD pattern of as-synthesized $FeSb_2$ particles was recorded in the 2θ range of 20°–90° at room temperature. The pattern shown in Fig. 1 indicates the crystallization of $FeSb_2$ in orthorhombic phase (marcasite-type) with space group Pnnm. The relative peak intensities and peak positions of generated pattern are consistent with thereferenced XRD pattern (ICCD No: 04-010-4959). The strong reflection peaks correspond to (011), (101), (120), (210), (111), (130), (211) and (031) planes. The sharp peaks without any significant broadening showed the highly crystalline nature of the sample. The presence of twosmall additional peaks at around 30° and 36° correspond to the Sb and Fe_3O_4 phase, respectively, suggesting minor impurity in the sample.

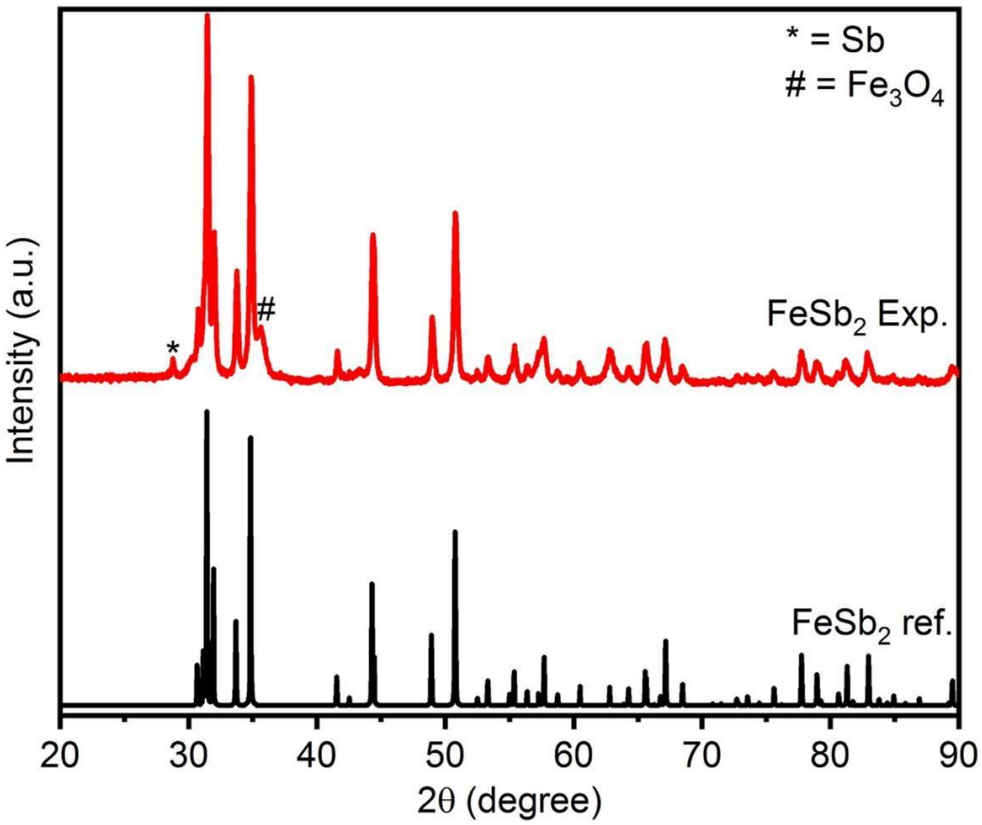

Fig. 1. XRD pattern of as synthesized $FeSb_2$ along with reference data.

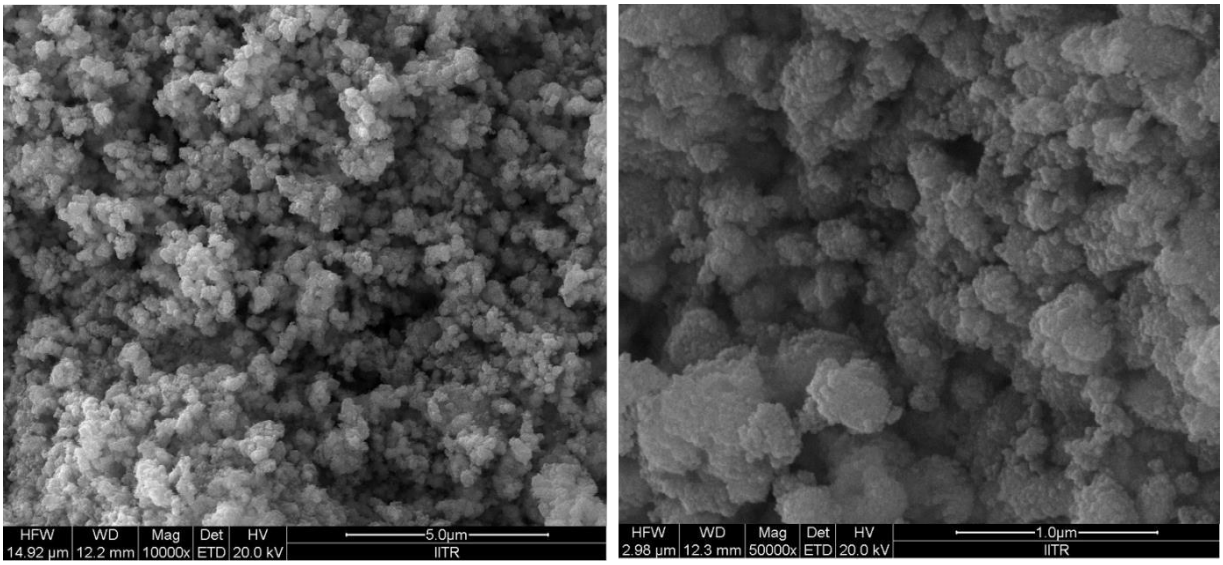

Fig. 2. SEM images of as-synthesized $FeSb_2$ particles.

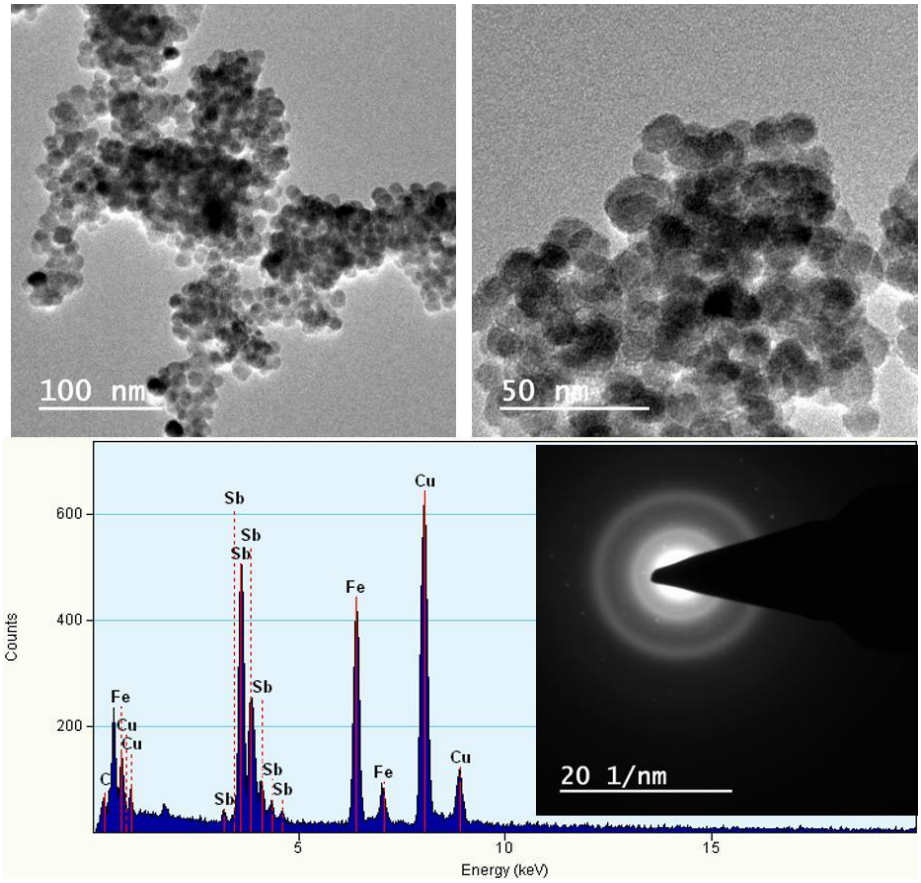

Fig. 3. TEM images of $FeSb_2$ smaple with EDXA analysis and SAED pattern.

SEM & TEM analysis: The scanning electron microscopy (SEM) images of $FeSb_2$

powder shown in Fig. 2 indicates the spherical morphology of the particles with very high agglomeration. The transmission electron microscopy (TEM) images of as-synthesized $FeSb_2$ were captured by drop-casting the ethanol dispered particles on a copper-grid and are shown in Fig. 3. The spherical morphology observed from TEM images is in good agreement with thatobserved from SEM images (though agglomerated). The size of the nanoparticles was found tobe in the range of 10-20 nm. The presence of bright rings in the selected area electrons diffraction (SAED) pattern, indicates that the synthesized particles are well crystalized and arepolycrystalline in nature. The elemental composition of Fe and Sb, based on Energy dispersiveX-ray analysis (EDXA) was found to be in the ratio of 1:1.23, confirming the formation of additional Fe_3O_4 phase as indicated by the XRD pattern.

Thermal analysis: Thermal analysis of $FeSb_2$ was investigated with a 10 mg sample withsame amount of reference alumina powder in argon atmosphere in the temperature range 35 - 1400 °C and the recorded data are shown in Fig. 4. The TGA (red) and corresponding first derivative DTG (blue) curve clearly indicate that about 22 % weight loss occurs at 748 °C

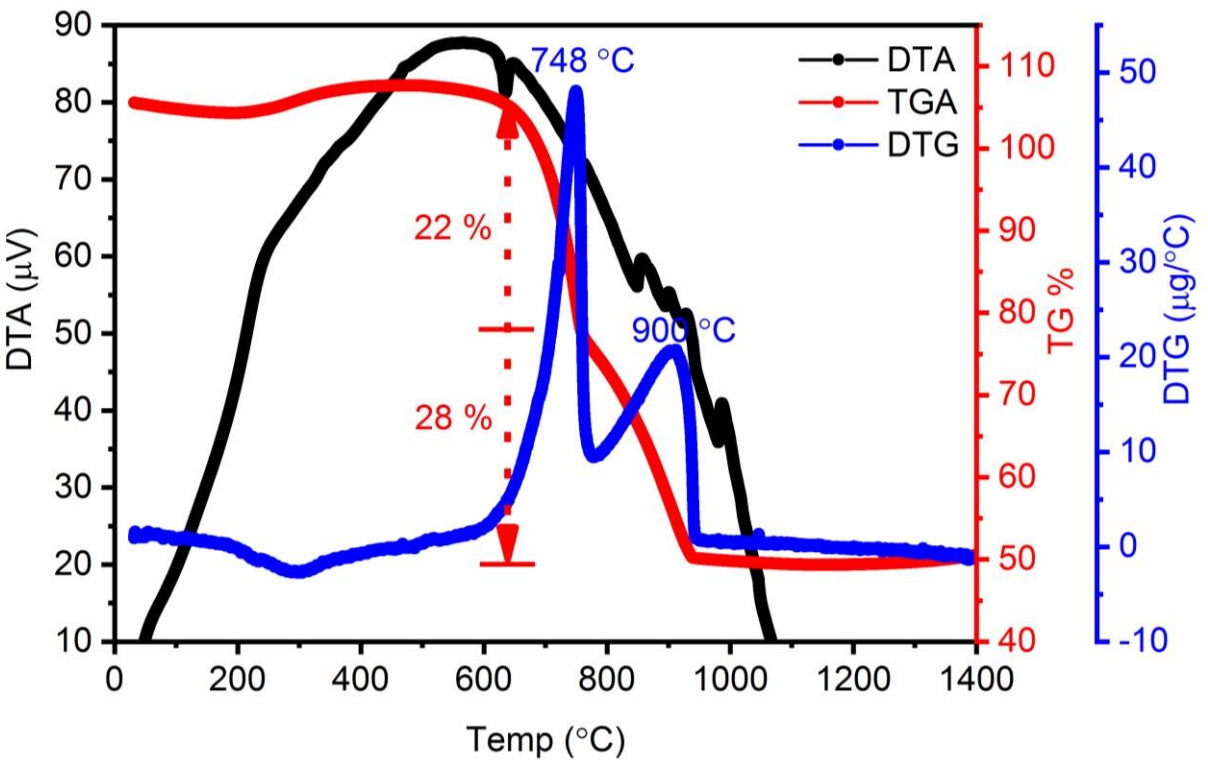

Fig. 4. Thermal analysis of $FeSb_2$ sample: TGA (red), DTG (blue) & DTA (black) recordedunder argon atmosphere.

Which corresponds to the melting temperature of $FeSb_2$. The approximately 28 % reduction inweight at 900 °C is probably due to decomposition of $FeSb_2$ into FeSb at this temperautre. Bothconclusions are supported by endothermic peaks in DTA (black) curve at the respective temperatures. No weight loss before the melting point of $FeSb_2$ suggests its anhydrous nature. The small amount of weight gain after 200 °C is probably due to oxidation or buoyancy effects.

Magnetic measurement: Fig. 4 shows the field dependence of the magnetization at fivedifferent temperatures in the range of 5-300 K. The magnetic moment increases with the appliedmagnetic field and magnetization saturation occurs at the magnetic field of ~3 KOe. The generation of hysteresis loop and greater opening at low temperature reflect the ferromagneticnature of the particles. The coercivities values of 240, 90, 40, 10 and 20 Oe, were observed at5, 30, 60, 150 and 300 K, respectively. Since, the pure $FeSb_2$ is diamagnetic at low temperatures

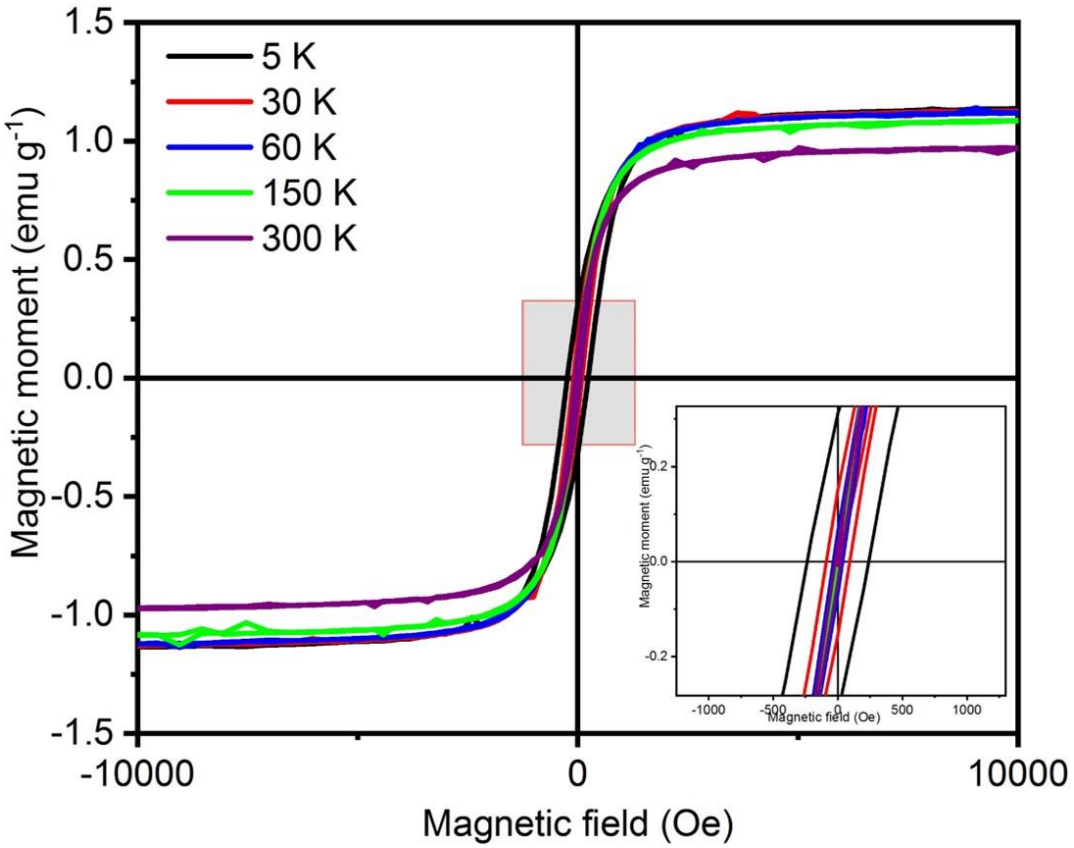

Fig. 5. Magnetization of as-synthesized $FeSb_2$ sample as a function of appliedmagnetic field at five different temperatures (5-300 K). Insets: enlarged area around the origin.

and paramagnetic at 300 K [13], it is evident that this weak ferromagnetism is due to the small Fe_3O_4 impurity in the sample.

Conclusion

We have presented a one-pot wet-chemical synthesis of $FeSb_2$ nanoparticles via simple polyol approach using metal chlorides as precursors. The particle size has been controlled under 20 nm without the addition of any external capping and reducing agent. Sample characteristics hasbeen analysed using XRD, SEM, TEM, TGA, DTA and SQUID. The result revealed a slight magnetic impurity of Fe_3O_4 in the sample. The synthesis method established here should guide the researchers to develop other binary intermetallic using transition-metal and main group elements.

REFERENCES

[1] Birkel, C. S.; Kieslich, G.; Bessas, D.; Claudio, T.; Branscheid, R.; Kolb, U.; Panthofer, M.;Hermann, R. P.; Tremel, W. Wet chemical synthesis and a combined X-ray and mossbauer study of the formation of $FeSb_2$ nanoparticles. *Inorg. Chem.* **2011,** 50, 11807-11812.

[2] McGrath, A. J.; Thompson, J. D.; Ronning, F.; Ivanov, S. A. One-pot size-controlled synthesis of tin- and antimony-based intermetallic nanoparticles. *Chem. Mater.* **2023,** 35, 457-467.

[3] Furukawa, S.; Komatsu, T. Intermetallic compounds: promising inorganic materials for well-structured and electronically modified reaction environments for efficient catalysis. *ACS Catal.* **2017,** 7, 735-765.

[4] Homes, C. C.; Du, Q.; Petrovic, C.; Brito, W. H.; Choi, S.; Kotliar, G. Unusual electronic and vibrational properties in the colossal thermopower material $FeSb_2$. *Sci. Rep.* **2018,** 8, 11692-11701.

[5] Sun, P.; Oeschler, N.; Johnson, S.; Iversen, B. B.; Steglich, F. $FeSb_2$: prototype of huge electron-diffusion thermoelectricity. *Phys. Rev. B* **2009,** 79, 153308-153312.

[6] Sun, P.; Oeschler, N.; Johnsen, S.; Iversen, B. B.; Steglich, F.; Narrow band gap and enhanced thermoelectricity in $FeSb_2$. *Dalton Trans.* **2010,** 39, 1012-1019.

[7] Zhao, H.; Pokharel, M.; Zhu, G.; Chen, S.; Lukas, K.; Jie, Q.; Opeil, C.; Chen, G.; Ren, Z.Dramatic thermal conductivity reduction by nanostructures for large increase in thermoelectric figure-of-merit of $FeSb_2$. *Appl. Phys. Lett.* **2011,** 99, 163101-163104.

[8] Kumari, L.; Li, W.; Huang, J. Y.; Provencio, P. P. Nanosize transition metal antimonides, NiSb and $FeSb_2$: solvothermal synthesis and characterization. *J. Phys. Chem. C* **2010,** 114, 9573–9579.

[9] Datta, A.; Nolas, G. S. Synthesis and characterization of nanocrystalline $FeSb_2$ for thermoelectric applications. *Eur. J. Inorg. Chem.* **2012,** 1, 55–58.

[10] Wei, K.; Martin, J.; Nolas, G. S. Synthesis, SPS processing and low temperature transportproperties of polycrystalline $FeSb_2$ with nano-scale grains. *Mater. Lett.* **2014,** 122, 289–291.

[11] Saleemi, M.; Tafti, M. Y.; Jacquot, A.; Jägle, M.; Johnsson, M.; Toprak, M. S. Chemical synthesis of iron antimonide ($FeSb_2$) and its thermoelectric properties. *Inorg. Chem.* **2016,** 55,1831−1836.

[12] Kieslich, G.; Birkel, C. S.; Veremchuk, I.; Grin, Y.; Tremel, W. Thermoelectric propertiesof spark-plasma sintered nanoparticular $FeSb_2$ prepared via a solution chemistry approach. *Dalton Trans.* **2014,** 43, 558–562.

[13] Pokharel, M.; Zhao, H.; Modic, K. A.; Ren, Z.; Opeil, C. Magnetic properites of hot- pressed $FeSb_2$. *IEEE Trans. Mag.* **2014,** 50, 2400304.