Available online at www.pelagiaresearchlibrary.com

Pelagia Research Library

Der Chemica Sinica, 2011, 2 (1): 70-78

Pelagia Research Library
ISSN: 0976-8505
CODEN (USA) CSHIA5

Magnetic and Electric Properties of gel grown crystals of bismuth Tri -Iodide BiI_3

T. K. Patil[1] and K. B. Saraf[2]

[1]*Smt G.G.Khadse Science, Arts and Commerce College, Muktainagar(India)*
[2]*Pratap College, Amalne (India)*

ABSTRACT

In the present investigation, crystals of bismuth tri-iodide (BiI_3) were grown by a simple gel technique using single diffusion method. The optimum growth conditions were established by varying various parameters such as pH of gel solution, gel concentration, gel setting time, concentration of reactant etc. Gel was prepared by mixing sodium meta silicate ($Na_2SiO_35H_2O$), glacial acetic acid (CH_3COOH) and supernant bismuth chloride ($BiCl_3$) at pH value 4.4 and transferred in glass tube of diameter 2.5 cm and 25 cm in length. The mouth of test tube was covered by cotton plug and kept it for the setting. After setting the gel, it was left for aging. After 13 days duration the second supernant potassium iodide (KI_3) of 1M concentration was poured over the set gel by using pipette then it was kept undisturbed. After 72 hours of pouring the second supernatant, the small nucleation growth was observed at below the interface of gel. The good quality hexagonal BiI_3 crystals were grown in 33 days. These grown crystals were characterized by Magnetic Succeptibility, Electrical Conductivity, EDAX & SEM.

Keywords :- Gel Grown Bii_3 Crystals, Magnetic Succeptibility, Electrical Conductivity, EDAX & SEM.

INTRODUCTION

Now a day, various crystals have been used in electronic industry for controlling the frequency of radio waves, optical property in polarizing microscopes, in microwave communication, in digital telephonic instrumentation, in wireless and optical communication, in electronic and photonic devices [1-7]. A systematic study of crystallization in gel begins with Leisgang's famous discovery of periodic crystallization in gels. This method has gained considerable attention because of its simplicity and effectiveness in growing single crystal of certain compounds. This method is an alternative technique to solution growth with controlled diffusion. This growth process is free from convection. This is purifying process, free from thermal strain [8, 9]. Crystal habit of various crystals, grown under different conditions and also by different methods were described by H. E. Buckley [10], P. Hartman [11], K. Kern [12], A. A. Chernor [13], W. K. Burton [14] and J. W. Mullin [15]. The various process parameters such

as degree of saturation, type of solvent [16], pH of the gel media [17, 18], presence of impurities [19] and the change in growth temperature also presumably affect significantly the morphology of the crystal [20].

In the present study, crystals of BiI_3 were grown by a simple gel technique using single diffusion method. The optimum growth conditions for crystals were obtained. These conditions were established & reported.

MATERIALS AND METHODS

Test tubes are used as crystallizing vessels. Sodium met silicate ($Na_2SiO_35H_2O$) gel was used as a growth media. Gel was prepared by glacial acetic acid and sodium metal silicate, having different pH values .The chemical used for growth of single crystals of Bismuth Iodide were Ch_3CooH, $Na_2Sio_35H_2o$, $Bicl_3$ or $Bi(No_3)_3$ KI all chemicals are of AR grade .Different molar masses were tried to determined the optimum growth conditions one of the reactant having different concentration were incorporated into gel. This solution was then transferred to borosil glass tube of diameter 2.5cm and 25cm in length (height). The mouth of the tube was covered by cotton plug. After setting of the gel it was left for aging for different periods of time other reactant having different concentrations was then added as supernant over the set gel. Experiments were carried out by changing different concentration of the reactants. The Chemical reaction inside the gel can be expressed as

$$XCl_3 + 3YI \rightarrow XI_3 + 3YCl \qquad Or$$

$$X(NO_3)_3 + 3YI \rightarrow XI_3 + 3YNO_3$$

Were X=Bi and Y=K or Na.

RESULT AND DISCUSSION

The optimum conditions for growth of bismuth Iodide crystals' are as shown in table (1) & effect of concentration of reactant on habit and size of Bismuth Iodide crystals are given in table (2).

Table (1) optimum conditions for growth of bismuth Iodide crystals

Sr. No	Conditions	Bismuth Iodate
1	Density of sodium metasilicate solution	1:04 gm/cm^3
2	Amount of 2N Acetic Acid	5ml
3	pH of gel	4.40
4	Temperature	Room temperature
5	Concentration of $BiCl_3$	0.5m, 1m
6	Concentration of KI	0.5m
7	Gel Setting time	13 days
8	Gel aging time	72hrs.
9	Period of growth crystal	33 days

Table (2) effect of concentration of reactant on habit and size of Bismuth Iodide crystals

Sr. No.	Concentration of reactant in gel	Concentration of reactant above gel	Remark
1	$Bicl_3$ 0.5m 5ml	KI or Na I 0.5m,15ml	Large no of micro crystals were produced. They were attached to themselves and form a thick larger of crystals at the interface crystals were transparent shining tinny (Smaller) in size there were no diffusion of crystals below interface
2	$Bicl_3$ 1.0m, 5ml	KI or Na I 0.5 m, 15ml	Large no of microcrystals were produced The shape of crystals appeared spherical and like a stones with high magnification color is reddish, white the crystals were opaque form a circular ring in test tube and day by day their smaller size remain constant.
3	$Bicl_3$ 1.5m, 5ml	KI or NaI 0.5m, 15ml	Large no of micro crystals produced but they are neigligible and the size of crystals are more smaller than previous cases.

Fig1. Crystals of Bismuth Iodide inside the test tube

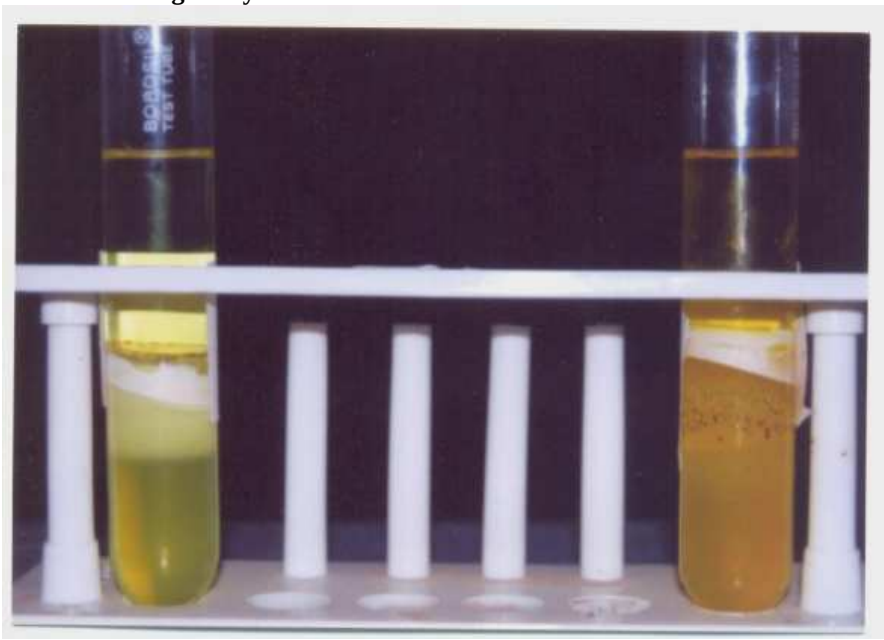


Fig 2. Few crystals of Bismuth Iodide

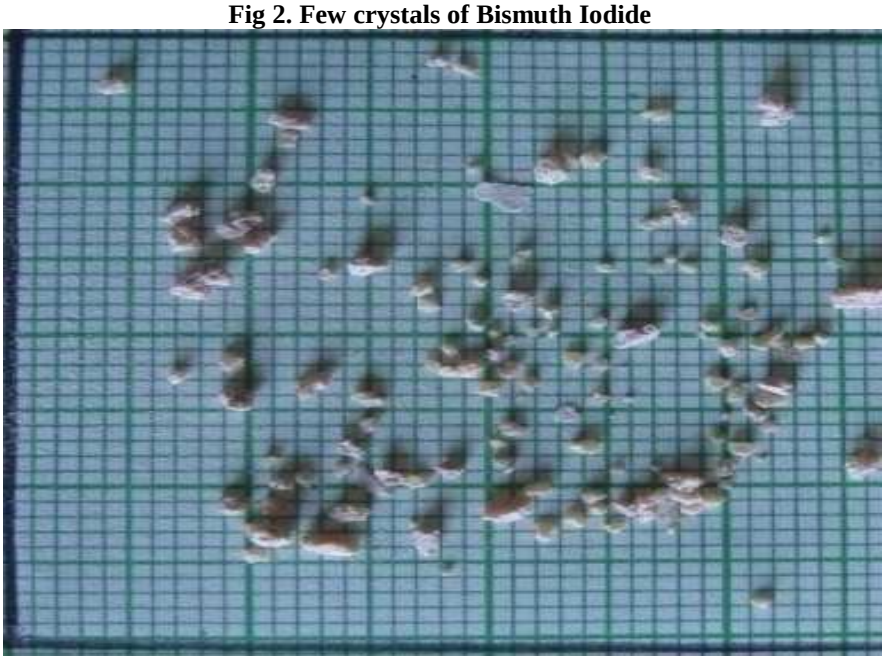


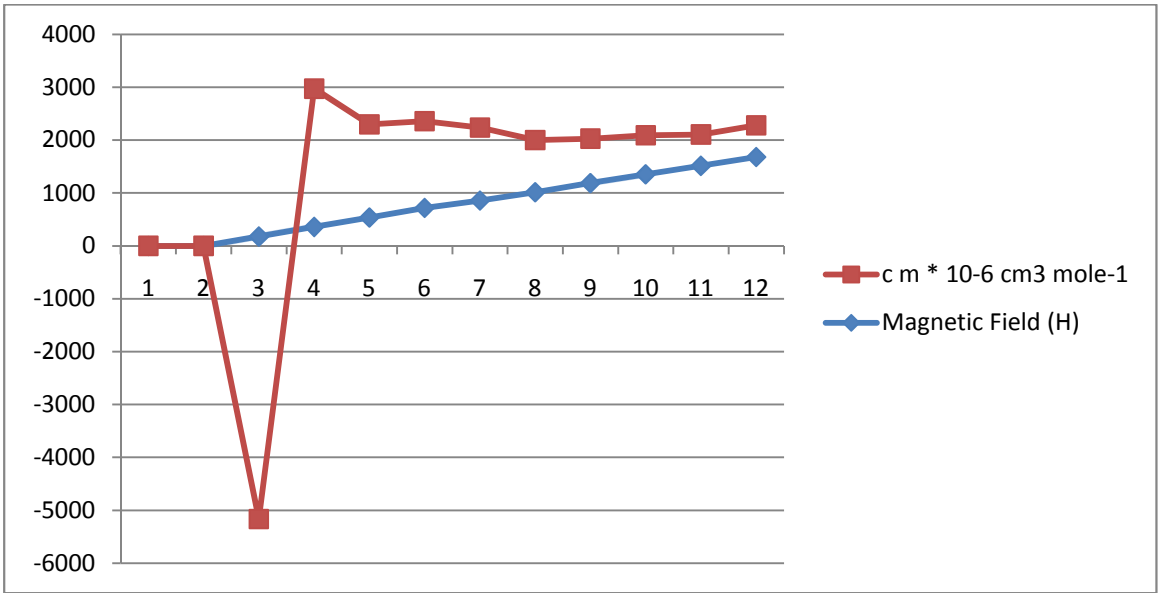
Fig. 1. Shows transparent crystals of Bismuth Iodide attached to themselves and forming a thick layer at the interface. fig 2. shows different habits with their scaling on a graph paper .Grown Bismuth Iodide crystals were characterized by XRD, FTIR, and the chemical percentage of component Bismuth and Iodine in BiI3 and is calculated and Examined.

Magnetic Susceptibility (Experiment for Bismuth Iodide)

Table (3): MAGNETIC SUCCEPTIBILITY of BiI_3

Sr. No	Current in A	Magnetic Field (H) Guass	Weight of sample in gm	Difference in wt m	$\chi_m * 10^{-6} cm^3 mole^{-1}$
1	0	0	4.694	----	0
2	0.2	178	4.695	- 0.001	-0.005342
3	0.4	360	4.692	+ 0.002	0.002612
4	0.6	537	4.691	+ 0.003	0.001760
5	0.8	718	4.689	+ 0.005	0.001641
6	1.0	859	4.688	0.006	0.001376
7	1.2	1016	4.688	+ 0.006	0.000983
8	1.4	1188	4.687	0.007	0.000839
9	1.6	1354	4.686	+ 0.008	0.000738
10	1.8	1515	4.686	0.008	0.0005900
11	2.0	1680	4.684	+ 0.010	0.0005997

Fig 3. Graph of Magnetic Field (H) Guass V/s $\chi_m * 10^{-6} cm^3 mole^{-1}$



Observations :-

a) Weight of empty holder + Holder Assembly (test tube) without magnetic field = 4.595 gm
b) Weight of empty holder + Holder Assembly (test tube) + sample powder without magnetic field = 4.694 gm
c) Weight of sample powder M= b – a = 4.694 – 4.595 = 0.099 gm

- m = Change in weight (m) of sample powder with magnetic field =0.002 gm
- L = Height of sample powder in test tube = 1.5 cm
- ρ = Density of specimen = 5.7 g/cm^3
- H = Applied magnetic field = 360 gauss (for 0.4 A current)
- M= Weight of specimen examine = 0.099 gm

- g = Acceleration due to gravity = 980 cm/sec^2

Formula :- The magnetic succeptibility (χ) of Bismuth Iodide (BiI_3) powder is given by relation.

$\chi = 2mgL\rho/MH^2 = 2 * 980 * 1.5 * 5.7 * 0.002 / 0.099 * (360)^2$

$\chi = 0.00261$

Electrical Conductivity of Bismuth Iodide BiI_3

OBSERVATIONS :- 1) Height of thickness of pallet = 0.580 cm
2) Diameter of the pallet = 0.950 cm
3) Radius of pallet = r = 0.475 cm =d/2
4) Voltage = 0.50 mv (constant)

K = l/RA K= l/Rπr^2 **(since A=πr^2) l** = 0.58 cm = 5.80 * 10 $^{-4}$ m **r** = 0.475 cm = 4.75 * 10 $^{-4}$ m**)**
K = 5.80 * 10 $^{-4}$ / R * 3.142 *(4.75 * 10 $^{-4}$)^2
K = 5.80 * 10 $^{-4}$ / R * 3.142 *(4.75)^2 * 10 $^{-8}$
K = 5.80 / R * 3.142 *(4.75)^2 * 10^{-4}
K = 5.80 * 10^4 / R * 3.142 *22.56 K = 5.80 * 10^4 / R * 70.8913
K = 0.0818 * 10$^{4/}$R K =8.18 * 10$^{2/}$R

Table (4): Electrical Conductivity of Bismuth Iodide BiI_3

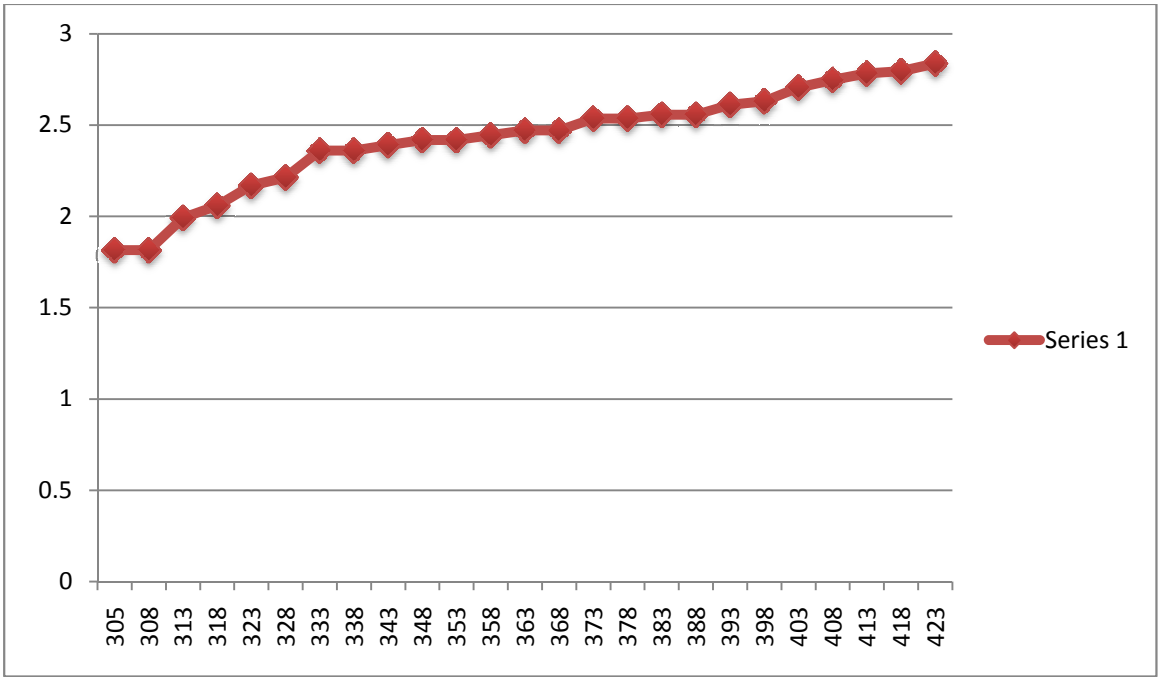
Sr. No	Temp T ^{o} k	1*10 $^{-4}$ /T	Current in A I * 10 $^{-4}$	Resistance R in Ω R * 10 $^{-4}$	Conductivity in mho/cm k* 10 $^{-4}$	Log K
1	423	23.64	0.42 4.2 * 10 $^{-4}$	01.19	687.39	2.83720321
2	418	23.92	0.38 3.8 * 10 $^{-4}$	01.31	624.42	2.795476805
3	413	24.21	0.37 3.7 * 10 $^{-4}$	01.35	605.92	2.782415288
4	408	24.50	0.34 3.4 * 10 $^{-4}$	01.47	556.46	2.747571383
5	403	24.81	0.31 3.1 * 10 $^{-4}$	01.61	508.07	2.705923552
6	398	25.12	0.26 2.6 * 10 $^{-4}$	01.92	426.06	2.629470763
7	393	25.44	0.25 2.5 * 10 $^{-4}$	02.00	409.00	2.611723308
8	388	25.77	0.22 2.2 * 10 $^{-4}$	02.27	360.35	2.556724526
9	383	26.10	0.22 2.2 * 10 $^{-4}$	02.27	360.35	2.556724526
10	378	26.75	0.21 2.1 * 10 $^{-4}$	02.38	343.69	2.536166896
11	373	26.80	0.21 2.1 * 10 $^{-4}$	02.38	343.69	2.536166896
12	368	27.17	0.18 1.8 * 10 $^{-4}$	02.77	295.30	2.470263447
13	363	27.54	0.18 1.8 * 10 $^{-4}$	02.77	295.30	2.470263447
14	358	27.93	0.17 1.7 * 10 $^{-4}$	02.94	278.23	2.444403956
15	353	28.32	0.16 1.6 * 10 $^{-4}$	03.12	262.17	2.418582994
16	348	28.73	0.16 1.6 * 10 $^{-4}$	03.12	262.17	2.418582994
17	343	29.15	0.15 1.5 * 10 $^{-4}$	03.33	245.64	2.390299089
18	338	29.58	0.14 1.4 * 10 $^{-4}$	03.57	229.13	2.36008196
19	333	30.03	0.11 1.1 * 10 $^{-4}$	03.57	229.13	2.36008196
20	328	30.48	0.10 1.0 * 10 $^{-4}$	05.00	163.60	2.2137833
21	323	30.95	0.09 0.9 * 10 $^{-4}$	05.55	147.38	2.16843855
22	318	31.44	0.07 0.7 * 10 $^{-4}$	07.14	114.56	2.059033
23	313	31.94	0.06 0.6 * 10 $^{-4}$	08.33	98.19	1.99206726
24	308	32.46	0.04 0.4 * 10 $^{-4}$	12.50	65.44	1.81584329
25	305	32.78	0.04 0.4 * 10 $^{-4}$	12.50	65.44	1.81584329

Calculations :- 1) I = 0.42 m A = 4.2 * 10 $^{-4}$ A
V = 0.5 mV = 5 *10 $^{-4}$ V

$R = V/I = 5 * 10^{-4} / 4.2 * 10^{-4} = 1.190\ \Omega$
$K = 8.18 * 10^2 / R = 8.18 * 10^2 / 1.19$
$K = 6.8739 * 100$
$K = 687.39$ mho/cm

2). $I = 0.38$ m A $= 3.8 * 10^{-4}$ A
$V = 0.5$ m V $= 5 * 10^{-4}$ V
$R = V/I = 5 * 10^{-4} / 3.8 * 10^{-4} = 1.31\ \Omega$
$K = 8.18 * 10^2 / R = 8.18 * 10^2 / 1.31$
$K = 6.2442 * 100$
$K = 624.42$ mho/cm

Fig 4. Graph of Temp T ^{o} k V/s Log K



EDAX :- Energy Dispersive Analysis is also called as elemental analysis by X ray (EDAX) In the present work elemental analysis of gel grown Bismuth Iodide, the NCL National Chemical Laboratory Pune fig (3) shows EDAX spectrum of Bismuth Iodide Table (5) shows the values of elemental content of the crystals as measured by the EDAX technique and the theoretical calculations from molecular formula. From the table it is clear that values of (wt %) and (At %) of BiI_3 in given crystals measured EDAX are close to with the estimated values calculated from molecular formula.

Table (5) for calculation of elemental analysis of gel grown Bismuth Iodide

Element	Content measured by EDAX		Content as calculated from molecular formula BiI_3	
	Wt %	At %	Wt %	At %
Bismuth	31.66 %	15.48	35.43 %	16.80
Iodide	58.83 %	54.63	64.55 %	48.82
	90.49		99.98	

Fig 6.1 Portion I

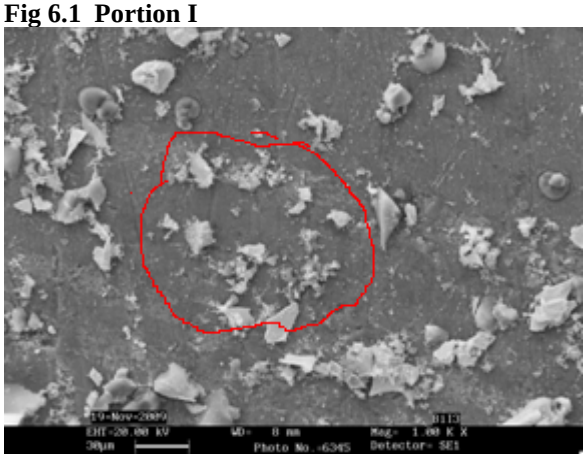


Fig 6.2 Portion II

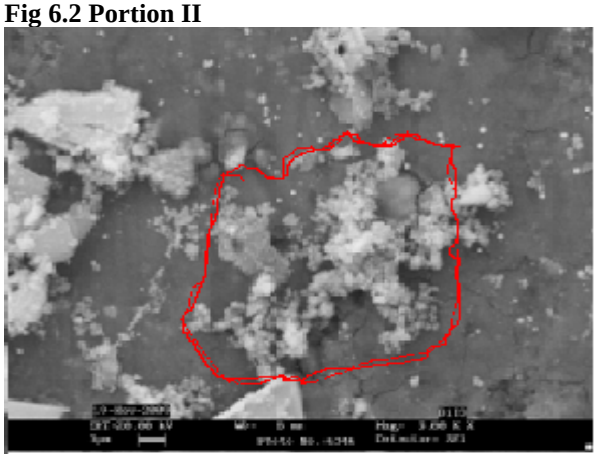


Fig 6.3 Portion III

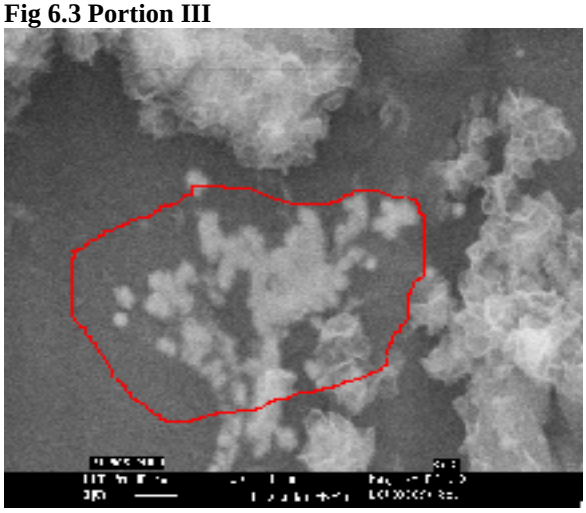


Fig 6.4 Portion IV

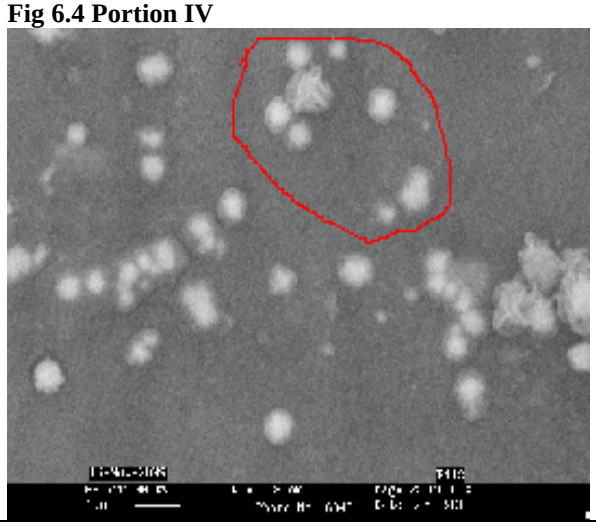


Fig 6.5

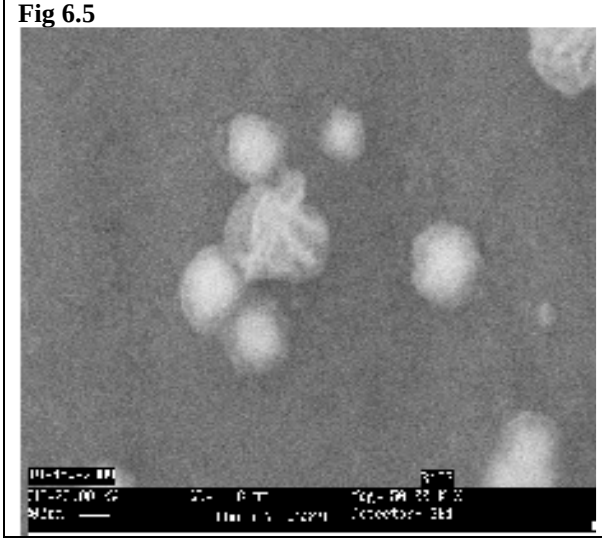


Fig 6.6

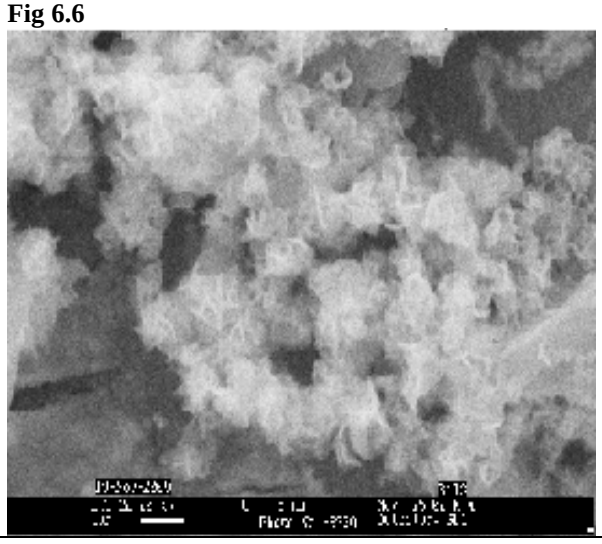
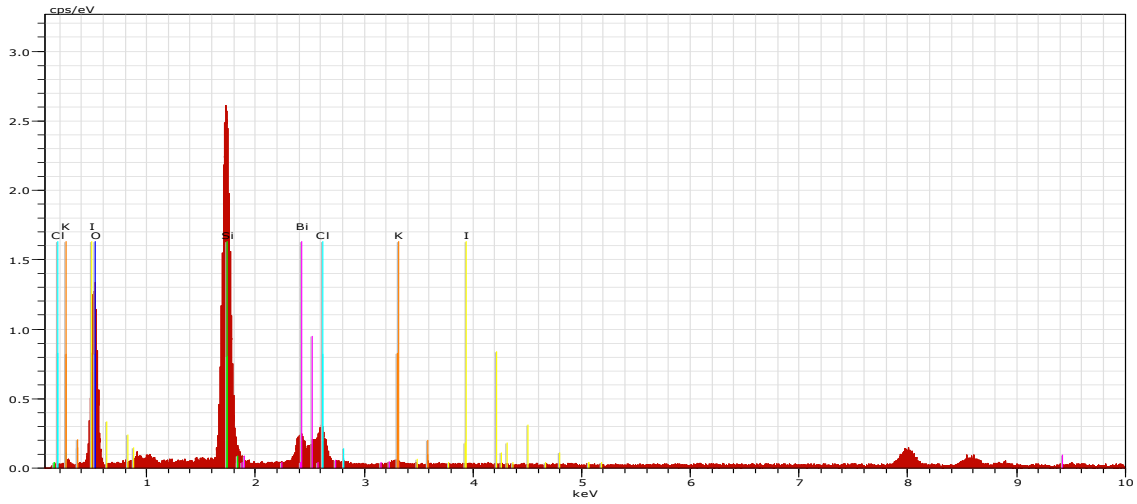


Fig 5 Energy Dispersive Spectrum of BiI_3



SEM :- Scanning Electron Microscopy of BiI_3

This technique combines of the resolution and analytical power with much ease of operation images can be formed from a very wide range of materials. From metal to ceramics and from semiconductor to polymers. These materials can be examined with low energy secondary electrons with high energy back scattered electrons or with other emission such as light, heat and sound. The high depths of field of the SEM images make it especially suitable for the study of the fracture, surfaces and complete microstructures such as those found in composite material.

In present work Scanning Electron Microscopy of powdered sample of gel grown Bismuth Iodide crystals was carried at NCL (National Chemical Laboratory) Pune fig (6.1) to (6.6) shows SEM images of the powdered sample of Bismuth Iodide. fig (6.1) shows the part of the crystal of Bismuth Iodide. It is observed that the face is in general dark, but which is covered with bright figures of different geometrical shapes randomly. Some of the bright figures have regular geometrical shape. Some of them are triangular in shape while others are pentagonal in shape

and some remaining is seen to be circular in shape. They do not have equal size and shape and randomly oriented through out the surface. The magnified version of part (I) is shown in figure (6.2), i.e. figure (6.2) shows that the various figures of part (I) from figure (6.1) are made up of circular grains having various size. Magnified version of part (II) from figure (6.2) is shown in figure (6.3), which manifest the large grain size of part (II) from figure (6.2). The enlarge portion of part (III) from figure (6.3)is shown in figure (6.4), which conformed spherical shape of the various grains of almost same size. Figure (6.5) shows enlarge portion of part (IV) of figure (6.5), which conformed the shape of the grain spherules.

CONCLUSION

From the above studies we observe that -

i. Magnetic measurement are importance in solving problems of molecular structure and bond type of the material. Offers, a means of detecting the presence of singly occupied electronic orbit. The value of magnetic susceptibility of BiI_3 closely related to theoretical ones. i.e. material BiI_3 is paramagnetic. Magnetic susceptibility is decreased as increase in temperature.
ii. The electrical conductivity of crystals closely related to chemical nature of compound the electrical conductivity increases as increase in temperature

iii. Gel growth technique is suitable for growing crystals of Bismuth iodide.
iv. Different habits of Bismuth iodide crystals can be obtained by changing parameters like gel density, gel aging, pH of gel, Concentration of reactants etc.
v. Crystals are quite transparent, and are of good quality.
vi. From EDAX the observed values well match with values calculated from molecular formula.
vii. From SEM the grain size of sample is spherical.

Acknowledgements
The authors are grateful to Prof.Dr.L.A.Patil, Head Department of Physics, Pratap College, Amalner, for providing laboratory facilities. Our special thanks to Department of Physical sciences, NMU, Jalgaon for providing Magnetic susceptibility & electrical conductivity facilities & authorities of NCL, Pune for EDAX & SEM facilities.

REFERENCES

[1] Garud, S. L. and Saraf, K. B., (**2008**) *Bulletin of Material Science*, 4, 639
[2] Garud, S. L. and Saraf, K. B., (**2009**) *Bulletin of Material Science*, 2, 187
[3] Blank, Z.j., (**1973**).*Crystal growth*, 18,281
[4] Bach, H. and Kuppers, H, (**1978**), *Acta Crystallography*, B34,263
[5] Armington, A, F, and O'Connor, j. j. (**1968**). *Journal of Crystal growth*, 3/4, 467
[6] Sangwal, K. and Patel, A. R. (**1974**). *J. Crystal growth*, 23, 282
[7] Joshi, M. S. and Trivedi, S. G. (**1983**). *Indian j. pure & App. Phys*., 21, 435
[8] Blank, Z. Brenner, W. and Okanoto, Y. (**1969**). *Material . Res. Bull*., 3,829
[9] Kurtz,S.K.and Perry,T.T. (**1968**). *J Appl .Phys*, 39,3798
[10] Morosin, B. Bergman, j. G. (**1973**), *Acta Crystallography*, B29, 1067.
[11] Blank Z. and Brenner,W. (**1969**) *Nature* ,222,79
[12] Patel A.R.and venkateshwara Rao. (**1978**) *A.J. Crystal Growth*, 43,351
[13] Nakamoto, K., (**1970**). *Infrared spectra of inorganic and coordination compounds (New York: John Wiley and sons inc) 2nd edition*
[14] Shitole, S. J. and Saraf, K. B. (**2001**).*Bulletin of Mate. Science* 5, 461
[15] Ranadive, D. Blank, Z, (**1969**), *Nature*, 223, 829
[16] X Sahaya shajan c (**2004**), *Bulletin of Mate. Science* ,4, 327
[17] Sharda Shitole & Suresh Kumar, (**2007**), *Bulletin of Mate. Science* 30, 349
[18] S. K. Arora & V. Patel, (**2006**), *Journal Of Physics*,28, 48
[19] P. Selvarajan, B.N.Das, (**1993**), *Journal Of Mate. Science* 12, 1210
[20] S. K. Arora & A.R. Patel, (**1976**), *Journal Of Mate. Science* 11, 843