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Magnetic and electric properties of gel grown crystals of bismuth iodate $Bi(IO_3)_3$

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ABSTRACT

In the present investigation, crystals of bismuth tri-Iodate [$Bi(IO_3)_3$] 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 Iodate (KIO_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 monoclinic $Bi(IO_3)_3$ crystals were grown in 36 days. These grown crystals were characterized by Magnetic Succeptibility, Electrical Conductivity, EDAX & SEM.

Keywords: Gel Grown $Bi(IO_3)_3$ 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 $Bi(IO_3)_3$ 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 Iodate were CH_3CooH, $Na_2Sio_35H_2o$, $Bicl_3$ or $Bi(No_3)_3$, (K IO_3) 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 in to borosil glass tube of diameter 2.5 cm and 25 cm 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

$XCl_3 + 3Y(IO_3) \rightarrow X(IO_3)_3 + 3YCl$ Or
$X(NO_3)_3 + 3Y(IO_3) \rightarrow X(IO_3)_3 + 3YNO_3$

Were X=Bi and Y=K or Na.

RESULTS AND DISCUSSION

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

Table (1) optimum conditions for growth of bismuth Iodate 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 K(IO_3)	0.5m
7	Gel Setting time	13 days
8	Gel aging time	72 hrs.
9	Period of growth crystal	36 days

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

Sr. No.	Concentration of reactant in gel	Concentration of reactant above gel	Remark
1	$BiCl_3$ 0.5m, 5ml	$K(IO_3)$ or Na (IO_3) 0.5m, 15ml	Large no of micro crystals were produced. They were attached to themselves and form a thick layer 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	$K(IO_3)$ or Na (IO_3) 0.5m, 15ml	Large no of micro crystals 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	$K(IO_3)$ or Na (IO_3) 0.5m, 15ml	Large no of micro crystals produced but they are negligible and the size of crystals are more smaller than previous cases.

Fig1. Crystals of Bismuth Iodate inside the test tube

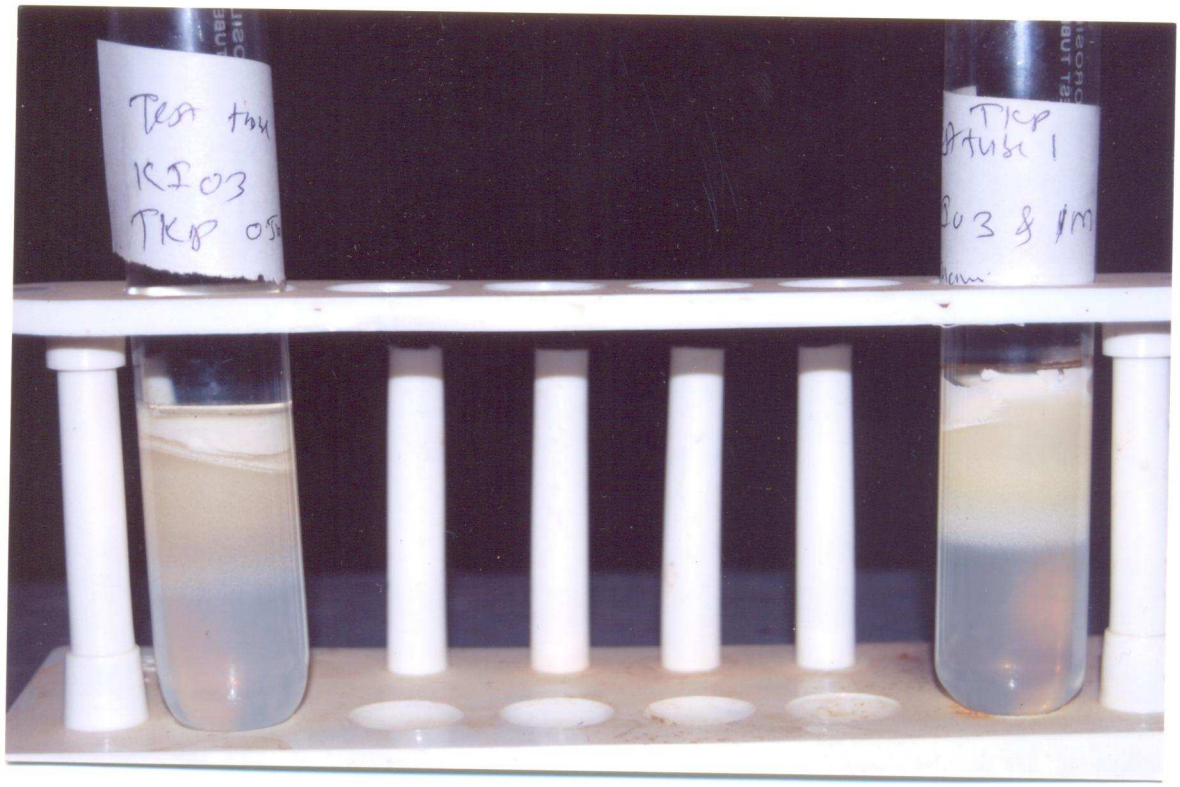


Fig 2. Few crystals of Bismuth Iodate

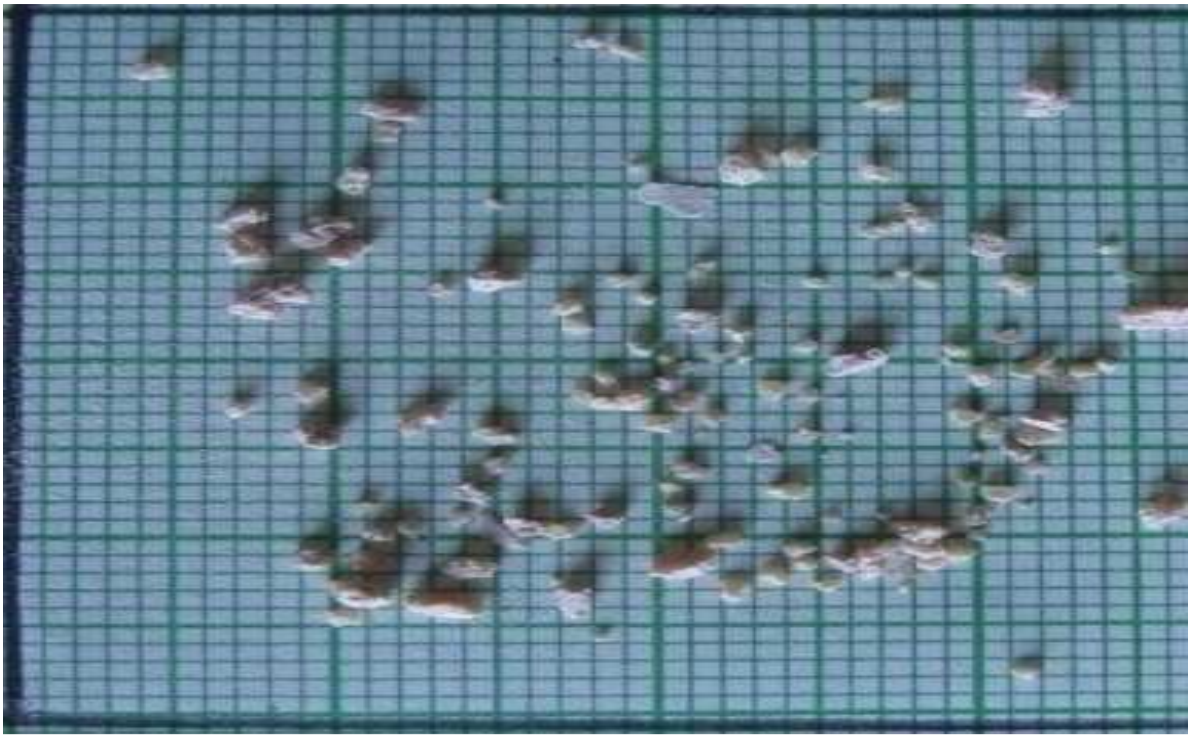


Fig. 1. Shows transparent crystals of Bismuth Iodate attached to themselves and forming a thick layer at the interface. fig 2. shows different habits with their scaling on a graph paper .Grown Bismuth Iodate crystals were characterized by Magnetic Susceptibility, Electrical Conductivity, EDAX & SEM.

MAGNETIC SUCCEPTIBILITY :- Experiment for Bismuth Iodate

Observation Table :- **(3) Magnetic Succeptibility of** $Bi(IO_3)_3$

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	4.647	----	0
2	0.2	173	4.648	- 0.001	- 0.005984
3	0.4	367	4.648	- 0.001	- 0.001329
4	0.6	538	4.646	0.001	0.0006188
5	0.8	712	4.646	0.001	0.0003533
6	1.0	894	4.645	0.002	0.0004482
7	1.2	1078	4.645	0.002	0.0003081
8	1.4	1177	4.644	0.003	0.0003878
9	1.6	1354	4.644	0.003	0.0002931
10	1.8	1518	4.643	0.004	0.0003109
11	2.0	1700	4.643	0.004	0.0002479

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 = 4.647 gm

c) Weight of sample powder M= b – a = 4.647 – 4.595 = 0.052 gm

- m = Change in weight (m) of specimen (sample powder) with magnetic field =0.001 gm
- L = Height of specimen (sample powder) in test tube = 0.6 cm
- ρ = Density of specimen = 7.92 gm/cm^3
- H = Applied magnetic field = 367 gauss (for 0.4 A current)

- M= Weight of specimen examine = 0.052 gm
- g = Acceleration due to gravity = 980 cm/sec^2

Formula :- The magnetic succeptibility (χ) of Bismuth Iododate $Bi(IO_3)_3$ powder is given by relation.

$$\chi = 2mgL\rho/MH^2$$

Calculation :- 1. $\chi = 2mgL\rho/MH^2$

I=0.8 A H=712 Gauss m=0.001 L=0.6cm

$\chi = 2 * 980 * 0.6 * 7.92 * 0.001 / 0.052 * (712)^2$

$\chi = 0.0003533$

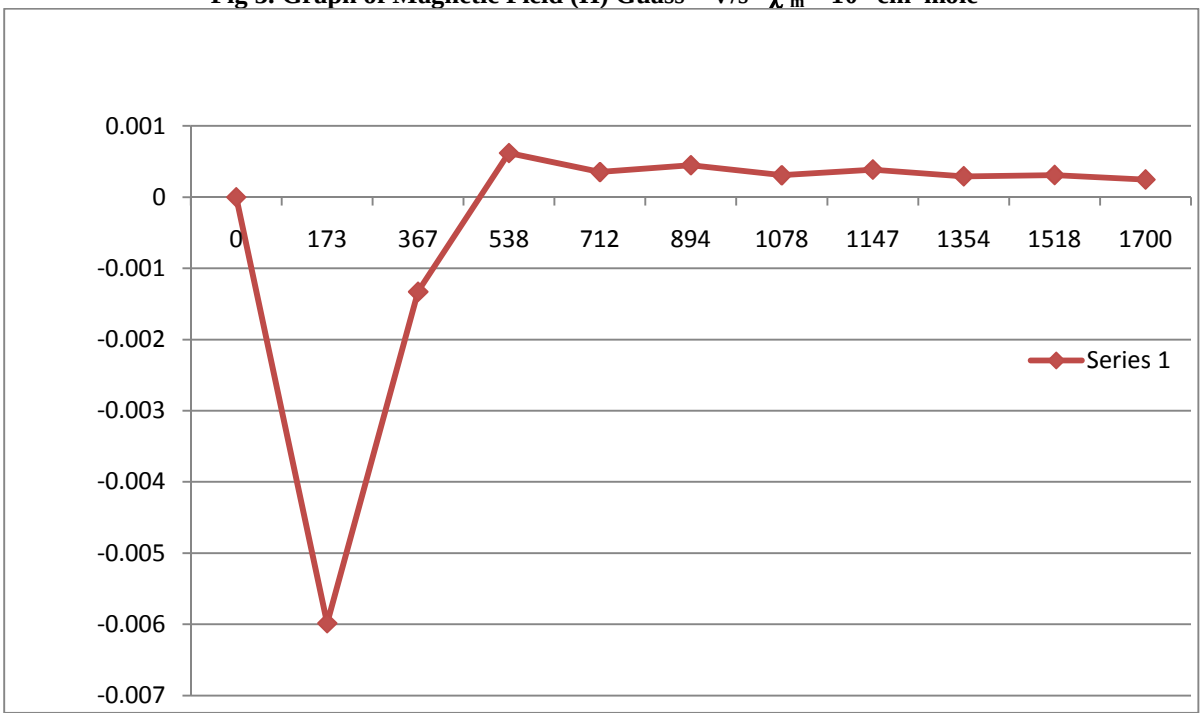
2. $\chi = 2mgL\rho/MH^2$

I=1.6 A H=1354 Gauss m=0.003 L=0.6cm

$\chi = 2 * 980 * 0.6 * 7.92 * 0.003 / 0.052 * (1354)^2$

$\chi = 0.0002931$

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



ELECTRICAL CONDUCTIVITY of BISMUTH IODATE $Bi(IO_3)_3$

Observations :- 1) Height / thickness (length) of pallet = 0.513 cm = 5.13 * 10^{-4}

2) Diameter of the pallet = 0.928 cm

3) Radius of pallet = r =d/2= 0.464 cm = 4.64 * 10^{-4}

4) Voltage = 0.50 mv (constant)

$K = l/RA/l \quad K= l/R\pi r^2$ **(since A=πr^2) l** = 0.513 cm = 5.13 * 10^{-4} m

r = 0.464 cm = 4.64 * 10^{-4} m**)**

$K = 5.13 * 10^{-4} / R * 3.142 *(4.64 * 10^{-4})^2$

$K = 5.13 * 10^{-4} / R * 3.142 *(4.64)^2 * 10^{-8}$

$K = 5.13 / R * 3.142 *(4.64)^2 * 10^{-4}$

$K = 5.13 * 10^4 / R * 3.142 * 21.53$ $K = 5.13 * 10^4 / R * 67.643$
$K = 0.0758 * 10^{4/}R$ $K = 7.583 * 10^{2/}R$

Calculations :-

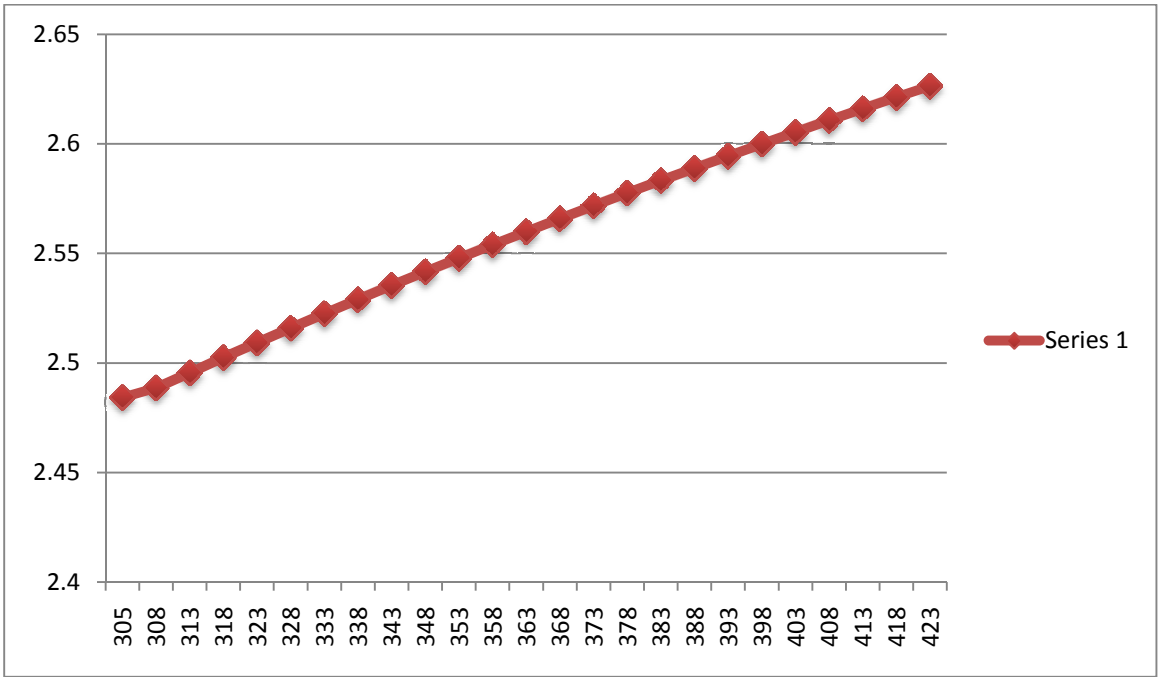
1) $I = 0.50$ m A $= 5.0 * 10^{-4}$ A
$V = 0.5$ mV $= 5 * 10^{-4}$ V
$R = V/I = 5 * 10^{-4} / 5.0 * 10^{-4} = 1.00\ \Omega$
$K = 7.583 * 10^2 / R = 7.583 * 10^2 / 1.00$
$K = 7.58300 * 100$
$K = 758.30$ mho/cm

2) $I = 0.49$ m A $= 4.9 * 10^{-4}$ A
$V = 0.5$ mV $= 5 * 10^{-4}$ V
$R = V/I = 5 * 10^{-4} / 4.9 * 10^{-4} = 1.02\ \Omega$
$K = 7.583 * 10^2 / R = 7.583 * 10^2 / 1.02$
$K = 7.4393 * 100$
$K = 743.43$ mho/cm

Observation Table :- (4) ELECTRICAL CONDUCTIVITY of BISMUTH IODATE $Bi(IO_3)_3$

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	0.50 $5.0 * 10^{-4}$	01.00	758.30	2.879841056
2	418	23.92	0.50 $5.0 * 10^{-4}$	01.00	758.30	2.871240082
3	413	24.21	0.50 $5.0 * 10^{-4}$	01.00	758.30	2.862804968
4	408	24.50	0.49 $4.9 * 10^{-4}$	01.02	743.43	2.862804968
5	403	24.81	0.48 $4.8 * 10^{-4}$	01.04	729.13	2.83475758
6	398	25.12	0.48 $4.8 * 10^{-4}$	01.04	729.13	2.826761353
7	393	25.44	0.45 $2.5 * 10^{-4}$	01.11	683.53	2.797052765
8	388	25.77	0.44 $4.4 * 10^{-4}$	01.13	671.06	2.782931043
9	383	26.10	0.41 $4.1 * 10^{-4}$	01.21	626.69	2.739959793
10	378	26.75	0.40 $4.0 * 10^{-4}$	01.25	606.64	2.673011686
11	373	26.80	0.36 $3.6 * 10^{-4}$	01.38	549.49	2.629419794
12	368	27.17	0.31 $3.1 * 10^{-4}$	01.61	470.99	2.596531122
13	363	27.54	0.28 $2.8 * 10^{-4}$	01.78	426.01	2.578753784
14	358	27.93	0.26 $2.6 * 10^{-4}$	01.92	394.94	2.41148502
15	353	28.32	0.25 $2.5 * 10^{-4}$	02.00	379.10	2.385677757
16	348	28.73	0.17 $1.7 * 10^{-4}$	02.94	257.92	2.3573821
17	343	29.15	0.16 $1.6 * 10^{-4}$	03.12	243.04	2.32715451
18	338	29.58	0.15 $1.5 * 10^{-4}$	03.33	227.71	2.26073902
19	333	30.03	0.14 $1.4 * 10^{-4}$	03.57	212.40	2.26073902
20	328	30.48	0.12 $1.2 * 10^{-4}$	04.16	182.28	2.18087105
21	323	30.95	0.12 $1.2 * 10^{-4}$	04.16	182.28	2.0283272
22	318	31.44	0.10 $1.0 * 10^{-4}$	05.00	151.66	1.87984106
23	313	31.94	0.07 $0.7 * 10^{-4}$	07.14	106.74	1.87984106
24	308	32.46	0.05 $0.5 * 10^{-4}$	10.00	75.83	2.879841056
25	305	32.78	0.05 $0.5 * 10^{-4}$	10.00	75.83	2.871240082

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



5.3 EDAX :- Elemental Dispersive Analysis by X rays (EDAX). Elemental Dispersive Analysis by X rays (EDAX) is used for the quantitative analysis.

In the present work elemental analysis of gel grown Bismuth Iodate crystals, was carried out at NCL National Chemical Laboratory Pune fig (5) shows EDAX spectrum of Bismuth Iodate. 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 Bi(IO3)3 in given crystals measured EDAX are close to with the estimated values calculated from molecular formula.

Fig 5 Energy Dispersive Spectrum of $Bi(IO_3)_3$

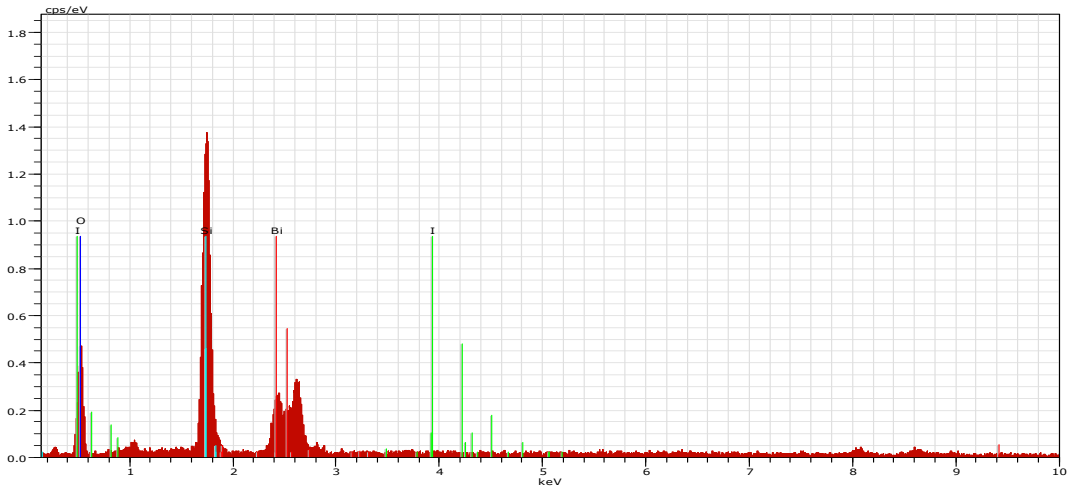


Table (5) for calculation of elemental analysis of gel grown Bismuth Iodate

Element	Content measured by EDAX		Content as calculated from molecular formula $Bi(IO_3)_3$	
	Wt %	At %	Wt %	At %
Bismuth	26.66	15.63	28.48	15.68
Iodine	47.54	59.45	51.88	60.46
Oxygen	22.42	24.58	19.64	23.86
	96.62		100.00	

5.4 SEM :- Scanning Electron Microscopy of Bismuth Iodate (Bi(IO3)3)

Fig 6.1

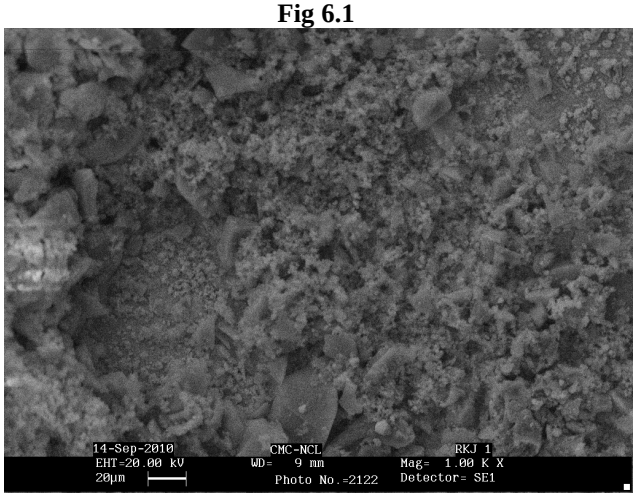


Fig 6.2 region (a)

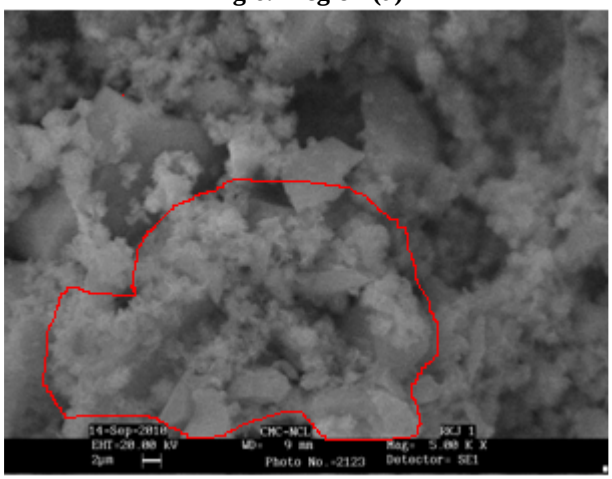
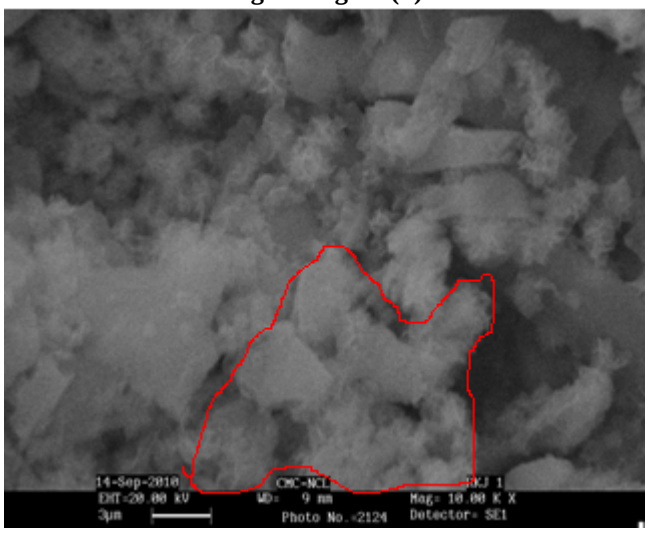


Fig 6.3 region (b)



In present work Scanning Electron Microscopy of powdered sample of gel grown Bismuth Iodate crystals was carried at NCL (National Chemical Laboratory) Pune and the successive photograph were taken at the magnification of 1.00, 5.00, 10.00 KX all the photographs were taken at common width 9 mm and EHT magnification 20 KV. And represented as Fig (6.1) to (6.3) shows SEM images of the powdered sample of Bismuth Iodate. Fig (6.1) is consider the whole surface is uniformly illuminated. i.e no difference of intensities means the surface is optically uniform. Fig 6.1 also shows mainly two types of geometrical figures it shows some pentagons of difference size with different orientations. It also shows large no of grains of different size. Fig 6.2 & Fig 6.3 shows parts of phase on higher magnification, if the regions on individual pentagons in Fig 6.1 are minutely observed. It also shows the well defined

boundaries with no attachment of micro crystals, as the surface is optically uniform & with no attachment of micro crystals these facts manifest, the growth conditions of crystals of Bismuth Iodate are somewhat controlled. If figs 6.2 & 6.3 are considered the region marked by a, from fig 6.2 is magnified as region b in fig 6.3 which supports the controlled growth conditions.

CONCLUSION

From the above studies we observe that -

- 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 $Bi(IO_3)_3$ closely related to theoretical ones. i.e. material $Bi(IO_3)_3$ is paramagnetic. Magnetic susceptibility is decreased as increase in temperature.
- The electrical conductivity of crystals closely related to chemical nature of compound the electrical conductivity increases as increase in temperature
- Gel growth technique is suitable for growing crystals of Bismuth iodide.
- Different habits of Bismuth Iodate crystals can be obtained by changing parameters like gel density, gel aging, pH of gel, Concentration of reactants etc.
- Crystals are quite transparent, and are of good quality.
- From EDAX the observed values well match with values calculated from molecular formula.
- From SEM the grain size of sample is spherical & pentagonal.

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