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GROWTH OF BARIUM IODATE CRYSTAL BY GEL TECHNIQUES

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Abstract:-In this review paper shows that the growth and study of single crystal barium iodate by gel technique. In this work there were three month data collected is shows by the 15 days intervals. By this method good control over nucleation, quality of crystal produces. The optimum conditions were established by varying various parameters such as pH of gel solution, gel concentration, gel setting time, concentration of the supernatant. Crystals having different Morphologies were obtained such as Prismatic, needle shaped, transparent, opaque needle. The characterization also studied using IR spectroscopy

Keywords: Barium iodate crystal, gel method, crystal growth .

INTRODUCTION

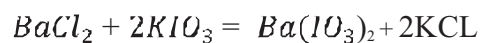
The gel techniques today, resting on a sound footing providing an tool for studying th kinematics and mechanism of bulk crystallization, as affected by various parameters such as temp, gel PH, gel density, gel aging, nature, purity, concentration of reactants and type of crystallization apparatus.

As the regard the mechanism by which crystals growth in the gels there have been evidence for two dimensional, spreading and pilling of growth layer taking place from one or more initiation centre. It can prove that the super saturation near the surface and homogeneous nucleation takes places.(Udea S.R. and Mullin J.M., Armington and O'Connar 1968; Blank and Brenner 1969; Blank 1973).

In the present work, single crystals of barium iodate $Ba(IO_3)_2$, were grown by gel technique . The optimum growth conditions for both the crystals were determined. As it has been observed that crystal habit is governed by kinetic rather than equilibrium condition (Joshi and Trivedi 1983), optimum conditions were established by varying various parameters such as gel concentration, pH of gel, gel aging time, concentration of reactants, concentration programming, effect of neutral gel, etc.

MATERIALAND METHODS

For the growth and study of single crystal barium iodide, various apparatus are used for gelling process such as digital PH meter, Magnetic stirrer, test tubes, burettes, pipettes Beaker etc. Growth of barium iodide is achieve by reaction of two components i.e barium chloride and potassium iodide.



For the preparation of gel – acid was taken in a beaker, to sodium matasilicate solution is added drop by drop with continues stirrer still PH is 4.2 . Then barium chloride is added with continuous stirrering. Then solution take place in test tube covered with cotton plug. The gel setting time is 15 day depending upon PH.

Potassium iodide (KIO_3) was used as feed the solution. Its different concentration varying from 0.2 to 0.4 were added over set gels.

RESULT AND DISCUSSION

The parameter which controls the nucleation and growth of single crystal are found to be

a) pH of gel : Gel of different pH value ranging 4 to 4.2 are recorded in table no.1. Higher pH values shows that proper crystal and deduce the number of nucleation. As the pH values are increase above 4.7 then gel set immediately which is leading to uncontrolled number nuclei and formation of too many microcrystals.

b) Concentration of supernatant -

0.2 M concentration (KIO_3) - less nucleation

0.4 M concentration (KIO_3) – greater nucleation and dendrites are observed
0.4 M concentration (KIO_3) – Large number crystal are observed at top most surface and less number crystal are observed at bottom.

a) Temperature: It is observed that habit of crystal modified due to the growth of temperature. But crystal growth at room temperature 22 to 30 °C

b) Effect of gel aging: To obtain the better quality of crystal and to reduced diffusion rate, gel aging is essential.

c) Optimum condition : The condition are follows

2 N Acetic acid : 4 CC

Density of sodium metasilicate solution: 1.04 gm/CC

Concentration of sodium Chloride solution: 0.4 M

pH of the mixture : 4.2

Temperature: 25 °C

Gel aging and setting Time 15 day

Observation:

Various concentrations of reactants have effects on the quantity of crystals. Tables 1 summarize the effects concentration of supernatant on quantity and type of barium iodide crystal. Figure 1 shows prismatic transparent crystals of barium iodide.

Table No.1 Effect of concentration of gel and supernatant on quantity of crystal

T.T. No	Na_2SiO_3 (ml)	Acetic acid	pH	KIO_3 (ml)	$BaCl_2$ (ml)	Description of crystal
1	4.4	4	10.5	0.2	5	No crystal
2	15	4.5	2.5	1.4	5	No crystal
3	15.5	5	2.5	0.4	5	No crystal
4	5	5	8.7	1	5	No crystal
5	4.5	4	5.24	2	5	3 needle 2 prismatic crystal
6	4	4	10.1	2	5	No crystal
7	4	4	11.1	2	5	No crystal
8	4	4	8.4	1.2	5	No crystal
9	4.5	4	4.6	1.5	5	No crystal
10	4.5	4	4.4	1	5	2 trans. prismatic crystal
11	3.1	4	4.9	1.2	5	No crystal
12	5	4	4.2	1.5	5	No crystal
13	6	4	4.2	2.5	5	No crystal
14	5	4	4.49	2	5	2 Needle prismatic crystal
15	5	4	6.7	1.5	5	No crystal
16	4.5	5	4.1	2	5	1 needle crystal
17	5	4	4.2	1	5	3 needle crystal
18	3.5	4	4.4	1.5	5	9 opaque needle crystal
19	4	4	4.04	1.5	5	1 needle crystal
20	3.5	4	4.2	1	5	Needle 7 prismatic crystal
21	4.5	4	4.2	1.5	5	2 needle crystal
22	4.5	4	4.1	1.5	5	1 needle crystal
23	4.5	4	4.2	1.5	5	1 needle crystal

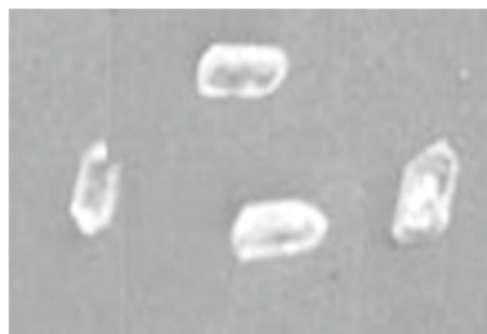


Figure 1. Prismatic transparent crystals of barium iodide

Characterization:

FT-IR is used for structural analysis. In the present investigation, spectra were obtained on Fourier Transform Infra Red spectroscopy (FT-IR) at SVPM's College of Pharmacy, Malegaon

Table No.2 show that Infrared (FT-IR) spectral analysis of barium iodide Figures 2 show FT-IR spectra of $\text{Ca}(\text{IO}_3)_2$. The spectra were scanned by placing sample KBr pellet in the sample beam in the range 500–4500 $1/\text{cm}$

The bands at 3000 $1/\text{cm}$ are due to O–H stretching and at 1568 $1/\text{cm}$ are due to H–O–H bending. Bands due to vibrations involving metal and iodine atoms are found near 582–802 $1/\text{cm}$. Fundamental infrared frequencies, observed in all iodide compounds in general, are also found in present FT-IR analysis, which confirms the iodide group of grown crystals.

Table No.2 Infrared (FT-IR) spectral analysis of barium iodide

No.	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	655.82	73.558	8.343	694.4	632.67	6.216	0.908
2	771.55	69.32	5.075	794.7	756.12	5.46	0.53
3	802.41	72.377	3.848	871.85	794.7	7.416	0.176
4	933.58	77.712	3.632	941.29	871.85	5.505	0.076
5	979.87	77.675	2.154	1003.02	941.29	6.36	0.379
6	1095.6	64.116	17.27	1303.92	1003.02	43.464	18.51
7	1419.66	66.443	14.705	1473.66	1357.93	14.422	3.988
8	1558.54	63.09	2.272	1566.25	1550.82	2.968	0.119
9	2360.95	79.517	6.249	2391.81	2345.52	3.336	0.463
10	3425.69	71.609	0.208	3433.41	3417.98	2.228	0.01



Fig.2 IR spectra of barium iodide

CONCLUSION:

Gel growth technique is suitable for growing crystals of barium iodate
Quantity of crystal are obtaining by changing the parameter such as pH of gel, Concentration of supernatant, Temperature, gel aging.
FT –IR spectra are confirms the iodide group of grown crystals

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