



## EFFECT ON FT-IR SPECTROSCOPY OF II GROUP OXALATE CRYSTALS GROWN BY GEL TECHNIQUE

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### ABSTRACT

As the gel medium prevents the turbulence and helps the formation of good crystal this gel technique has gained considerably. Starting from the agar gel solution, observed the growth mechanism of II group oxalate such as Barium oxalate and optimize the growth parameters. In the single diffusion, for different growth parameters, the platy, prismatic and needle shape transparent barium oxalate crystals were observed but no such result was observed by changing the reactant and supernatant. The double diffusion increase the size of barium oxalate but there is no change in the morphology of crystals with respect to those were observed by single diffusion. The FT-IR techniques were used to characterize the gel grown barium oxalate crystals.

**KEYWORDS:** Agar gel method, barium oxalate, single diffusion, double diffusion.

### INTRODUCTION

Crystals play a major role in body. In the human body many minerals are present in the dissolved forms. The body fluids contain minerals at various levels of saturation. When this body fluid gets supersaturated by the minerals, the crystallization takes place. These crystals have both beneficial as well as diseases effects on human body. The formation of teeth and bones are the beneficial effect, whereas the bio-crystallization are the root causes of several diseases in humans<sup>[1]</sup>, the common examples are urinary stones, Bone diseases, gall bladder stones, heart diseases<sup>[2]</sup> etc. The single crystals are also the backbone of the modern technology. The impact of single crystals is clearly visible in the industries like semiconductor, optics, acoustics, jewellery, medical applications, etc. As oxalates are insoluble in water and decompose before melting hence hydro silica gel<sup>[3]</sup> used for growing perfect crystals with minimum impurities and imperfection.

The mixed rare earth oxalates including cerium neodymium oxalate<sup>[4]</sup>, lanthanum copper oxalate<sup>[5]</sup> and cerium lanthanum oxalate crystal<sup>[6]</sup> have been grown by gel methods. By using this gel method the barium oxalate dehydrate<sup>[7]</sup>, the lead oxalate<sup>[8]</sup>, barium oxalate<sup>[9]</sup>, mixed barium ammonium oxalate<sup>[10]</sup>, barium copper oxalate<sup>[11]</sup>, neodymium copper oxalate<sup>[12]</sup> and calcium oxalate<sup>[13]</sup> has been reported

using gel. The single crystals of strontium tartrate tetra hydrates and trihydrates<sup>[14-20]</sup>, lithium doped strontium tartrate tetra hydrate<sup>[21]</sup> has been reported. The pure and Nickel doped strontium tartrate tetra hydrates single crystals were grown in silica gel.<sup>[22]</sup>

This paper describe the experimental procedure to grow the single crystals of II group oxalates such as barium oxalate using agar gel at ambient temperature by single and double diffusion techniques. After crystallization observe the effect on FTIR spectroscopy of this these techniques.



### MATERIAL AND METHODS

The principle involved in this method is very simple. The solutions of two suitable compounds which give rise to the required insoluble crystalline substance by mere chemical reaction between them are allowed to diffuse into the gel medium and chemically react as, AX and BY are the solutions of two compounds, due to its reaction gives rise to the insoluble or sparingly soluble substance AB and a waste product XY which is highly soluble in water.<sup>[1]</sup> For to grow the crystals, such reactions should be carried out in control manner by using gel. In the present work agar-agar gel was used in different concentration such as 0.5, 0.6, 0.7, 0.8, 0.9, 1.0 and 1.5% to grow barium oxalates.

### Single Diffusion

In this single diffusion technique, barium chloride solution of concentration 0.5M to 1M, were used as first reactants. While the solution of oxalic acid in concentration 0.5M, 0.75M, 1M & 2M were used as second reactant. For the experiment single glass tubes of 2.5 cm in diameter and 20 cm in height were used as the crystallization vessels. The same experiment was repeated by reversing the reactants and supernatant for the growth of barium oxalate.

**Double Diffusion:** For to grow barium oxalates using double diffusion method, 1.5% agar gel solution was transferred in U-glass tube of a diameter 2.5cm up to appropriate height and kept for setting. After setting and ageing of gel, slowly pour 1M concentration of  $BaCl_2$  solution in one limb of U tube, and 1M concentration of oxalic acid in other limb.

### RESULT AND DISCUSSION

In single diffusion:- After setting the gel, dendritic growth was observed below the surface during 5 to 6 days. Then after 8 to 10 days, single nucleation was observed at middle and lower side of test tubes for barium oxalate. The complete growth of barium oxalate

crystals was near about 6 to 8 weeks as shown in Fig.1.



**Figure 1: Good quality transparent, platy, needle shaped barium oxalate crystal.**

After reversing the reactants and supernatants solution of 1M barium chloride and 1M oxalic acid, it was found that the growth morphology was changed. Star type dendritic growth was seen in the test tube as shown in fig. 2.



**Figure 2: Star type dendrites crystals of barium oxalate.**

**In double diffusion:-** The total period of 4 to 6 week were taken for the growth of barium oxalate crystals in double diffusion. Good quality and large size

transparent platy shaped, needle shaped and dendrite crystals were obtained as shown in fig 3.





Figure 3: Good quality and large size transparent needle shaped and dendrite barium oxalate crystals.

Optimum conditions for the growth of barium oxalate crystals are summarized as tabular form as shown in table 1.

Table 1: Optimum conditions to grow barium oxalate crystals.

Condition	Single diffusion	Double diffusion
% of gel	0.9	1.5
Con.of barium chloride	1M	1M
Con.of oxalic acid	1M	1M
Gel setting period	2 days	2 days
Gel aging	3 days	3 days
Period of growth	60 days	60 days
Temperature	Room temp.	Room temp.
Quality	Less transparent	Good transparent
Shape	needle and platy shaped	needle and platy shaped
Size	9mmx2mmx 1.5mm	18mmx2mmx2mm

### CHARACTERIZATION

FTIR spectrum of the synthesized barium oxalate crystals using single and double diffusion technique was recorded in the range of 450 to 4000  $\text{cm}^{-1}$  is presented in Fig 4 and Fig 5. The broad envelope extending from 2800 to 3600  $\text{cm}^{-1}$  is assigned to the symmetric and asymmetric stretching modes of the water molecules. Considerably pronounced peak at 1650.02  $\text{cm}^{-1}$  in

single diffusion experiment, while 1652.68  $\text{cm}^{-1}$  in double diffusion, is assigned to asymmetric stretching vibrations of C=O groups of the  $\text{C}_2\text{O}_4^{2-}$  is related to O-H bending vibrations.<sup>[9]</sup> The IR band from 1377.44  $\text{cm}^{-1}$  & 1377.49  $\text{cm}^{-1}$  (Fig 3 & Fig 4) corresponds to the asymmetric stretching mode of C-O bond.<sup>[23]</sup> The sharp peak at 722.58  $\text{cm}^{-1}$  & 722.39  $\text{cm}^{-1}$  may be attributed to metal-oxygen (M-O bond).<sup>[9]</sup>

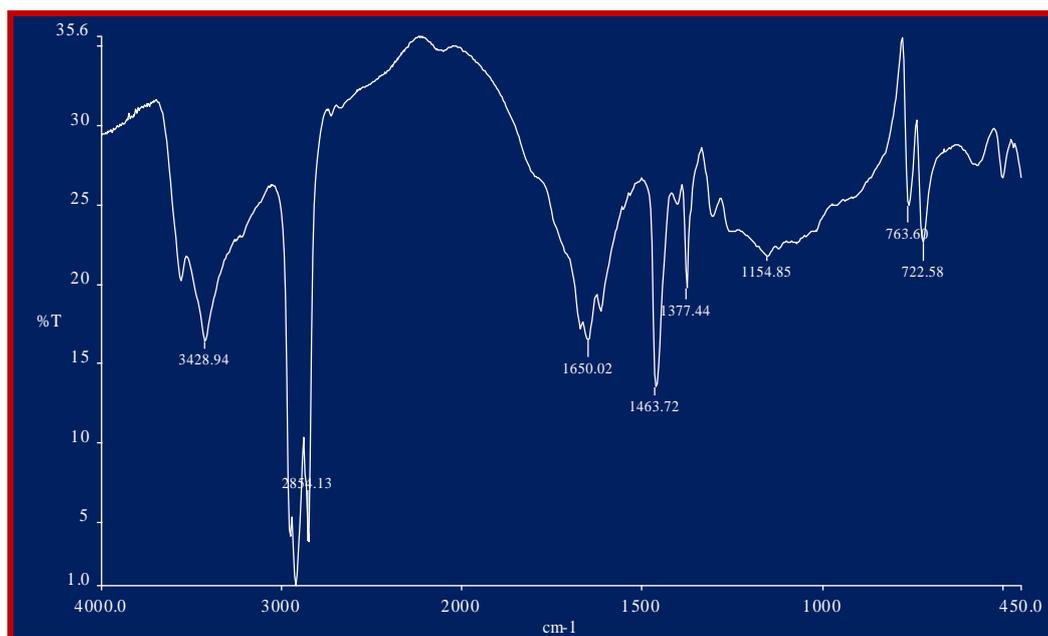


Figure 4: Infrared spectra of barium oxalate crystals grow in single diffusion techniques.

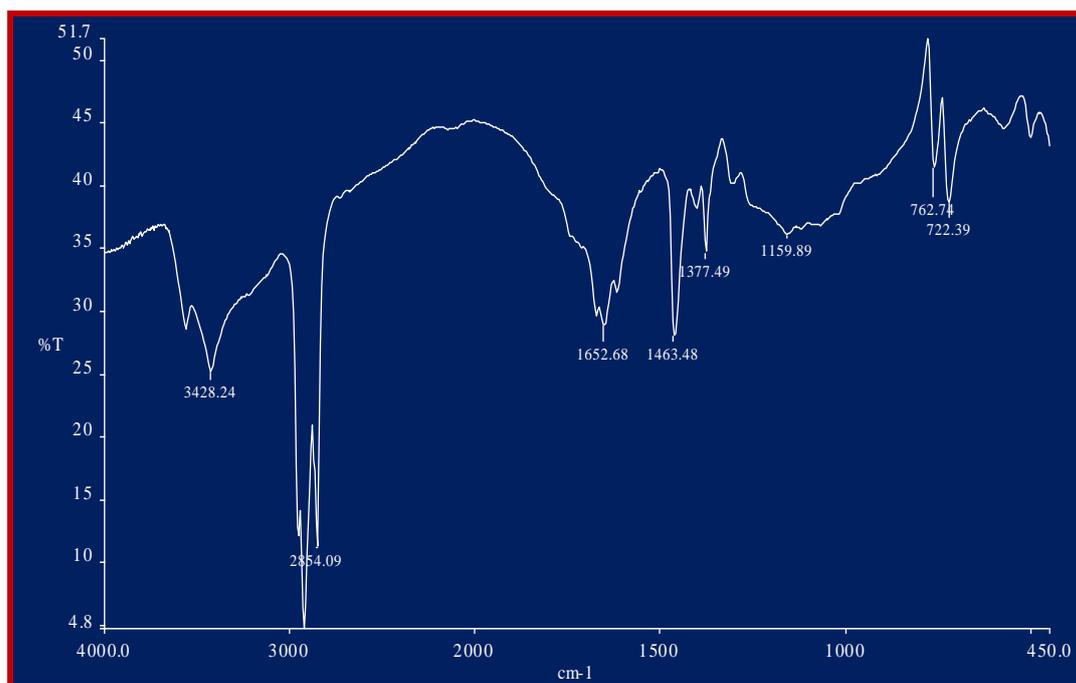


Figure 5: Infrared spectra of barium oxalate crystals grown in double diffusion techniques.

FTIR spectroscopic analysis of barium oxalate single crystals confirms the presence of functional groups associated with the oxalate ligands and the metal–oxygen bond. Assigned vibrational band with observed wave

numbers are summarized in Table 2. Table 2 shows that there is no difference in wave numbers of barium oxalate crystals grown by these two techniques.

Table 2: Assignment wave numbers and vibrations of barium oxalate crystals.

Bonds/Vibrational assignments	wave number (cm <sup>-1</sup> )	
	Observed in single diffusion	Observed in double diffusion
symmetric stretching bond of O-H	3428.94	3428.24
asymmetric stretching bond of O-H	2854.13	2854.09
asymmetric stretching of C=O	1650.02	1652.68
asymmetric stretching of C-O	1377.44	1377.49
Metal-oxygen bond	722.58	722.39

## CONCLUSION

The good qualities barium oxalate crystals of maximum size 9mmx2mmx1.5nm crystals were observed in single diffusion method whereas, 18mm x 2mm x 2mm were observed in double diffusion. FT-IR techniques have confirmed the crystalline nature of barium oxalate. The obtained wave numbers and vibrations of grown crystals were same in both techniques. The FTIR study reveals that the presence of metal oxygen bond. On the basis of the optimum conditions obtained for the growth of barium oxalate crystals, gel method with double diffusion technique is good for to growth such crystals.

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