Physical and Structural Characterization of Cadmium Oxalate Crystal Growth by Agar - Agar Gel Method

A.A.Gayakwad[1], S.J. Nandre[2], H. S. Pawar[3], A.J. More[4], R. R. Ahire[5]

[1]R.C. Patel Anu. Higher Secondary Ashram school,Shirpur,(Dhule) M.S.
[2]Department of Physics, UttamraoPatil Arts and Science College, Dahiwel, (Dhule) M.S.
[3]V.J.N.T. Late DalpatbhauRathod Junior College, Mordadtanda, (Dhule) M.S.
[4]Department of Physics S.G. Patil Art's, Commerce and Science College, Sakri (Dhule) M.S.

Abstract: Single crystal of cadmium oxalate crystal was grown by using agar-agar gel method at ambient temperature. The effect of different parameter on growth of cadmium oxalate crystals were studied and reported. The study of FTIR spectrum shows the presence of different bands of functional group. X-Ray diffraction analysis was done to determine the structure of grown crystals are crystalline in monocline system. The SEM photograph observed that crystals were grown by layer deposition.

Keywords: Gel method, XRD, FTIR, SEM.

1. INTRODUCTION

Crystal has always fascinated the mind and crystal growth has long been one of the most fascinating areas of research. Crystal growth is interesting field where the atomic nature of matter is clearly established through laboratory observation. Crystal growth with favourable physics and chemistry, crystal characterization with more advance devices, and their conservation into useful device play an important role in science and technology [1-2]. Single crystals are one of the most important groups of materials because single crystals have a uniformly continuous and highly order structure. Advance in crystal growth were in high demand given recent advance in the field of amplifier, transducers, semiconductor, ferrites, piezoelectric, photosensitive material, microelectronic and microprocessor [3]. Single crystal is most important part of modern technology with many applications such as advance display, catalysis, and permanent magnet [4] and optics [5]. It has widely used in the field of ceramics, physics, chemistry, metallurgy, medicine, and engineering [6]. Crystal growth in gel is simple and best method for growing single metal oxalate, transition metal oxalate [7-8] due to their solubility in water [9]. There are various techniques for growing crystal-like gel method, melt growth, vapour phase [10]. The gel technique is superior due to its simplicity, inexpensive, low cost as well as ph independent [11] and crystal can be grown at ambient temperature so many research attracted to gel method for crystal growth. By using silica gel method many research grown cadmium oxalate crystal [12-14] and very few researchers studied grown cadmium crystal oxalate using agar-agar gel method [15]. The cadmium ions have attracted much interest because of its coordination geometry and simple chemistry. Cadmium oxalate crystal is an extraordinary compound. Cadmium oxalate utilized in production of inks, paints, lithographic plate, photographic chemicals and used as a mordant in dyeing. There, during the present investigation it has been decided to synthesis and characterized cadmium oxalate crystal by using agar-agar gel method. Characteristics of cadmium oxalate crystals grown by various techniques are reported.

2. MATERIAL AND METHODS

In the present work to synthesis cadmium oxalate crystal was accomplished using gel diffusion technique. Cadmium chloride ($Cdcl_2$), oxalic acid ($CH_2C_2O_4$), and agar- agar powder was used as chemical compound and all chemical compounds were AR grade. In single diffuse method consist of single glass tube having 15 cm length and 2.5 cm diameter. 250 ml glass beaker was used as crystallizing vessel. The all the experimental solution prepared by using distilled water. The test tubes were filled by cadmium chloride of desire volume and molarity. The hot agar -agar solution was transferred to the containing solution of cadmium chloride and thoroughly mixed. This homogenous solution kept 24-48 hours for setting at ambient temperature in dust free atmosphere. After setting and aging of gel, the aqueous solution of oxalic acid with desire volume and molarity added over the set gel along the walls of test tube. The oxalate ions diffuse pass through the narrow pores of gel and react with cadmium ions present in the gel and formed cadmium oxalate crystals. The following reactions was expected to occur cadmium oxalate crystal were express as

$$CdCl_2 + H_2C_2O_4 \longrightarrow CdC_2O_4 + 2HCL$$

3. RESULT AND DISCUSSION

Optimal control parameters leading to good cadmium oxalate crystal growth were obtained. The different parameter such as concentration cadmium chloride and oxalic acid solution, percentage of gel and aging period have significantly affected the growth rate. It was observed that, the amount of concentration of cadmium chloride was increased, the size, shape and number of crystals were increased and other side percentage of agar gel increased the size and shape of number of crystals decreased. It was also observed that the rate of growth was fast at 24 hours than other. The grown cadmium oxalate crystal in single test tube as shown in fig.1.Good quality of grown single crystal having diamond, quadrilateral, panty, emerald, aquamarine shapes were obtained as shown in fig.2.The optimum condition for growing good cadmium oxalate crystals is shown in table 1.

Parameter	Values
Molarity of cadmium chloride	1M
Molarity of oxalic acid	1M
Percentage of gel	1%
Gel aging period	24 hours
Period of growth	25 days
Temperature	Ambient temperature
Nature and colour	Translucent and white

Table 1. Optimum condition for growth of cadmium oxalate crystals

Fig.1. Grown Cadmium Oxalate crystals in test tube

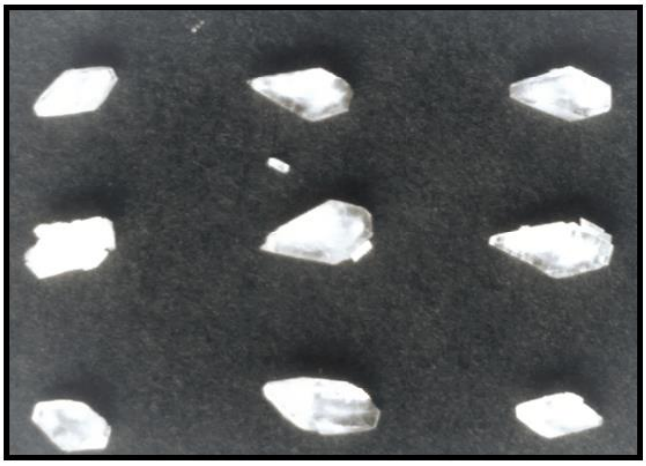

Fig.2.Different Morphology harvest Cadmium Oxalate Crystals

4. CHARACTERIZATION

4.1. X-ray Diffractometer (XRD)

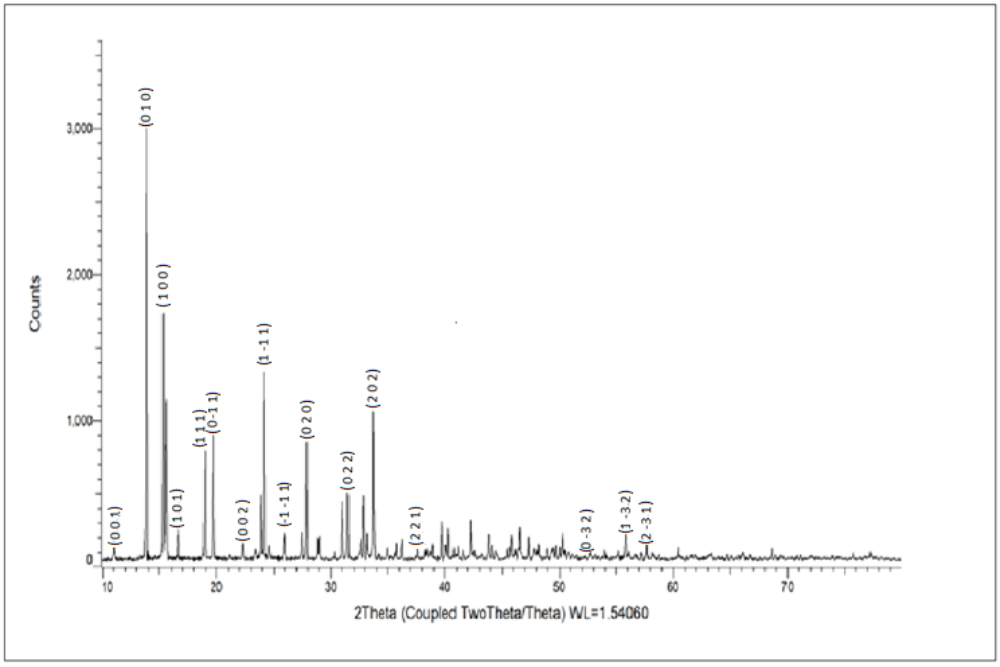

Figure 3. XRD pattern of the grown cadmium oxalate crystals

The powder x-ray diffraction (XRD) is one of best technique used for to determine physio-chemical made up of unknown materials. The crystal structure of cadmium oxalate crystals was studied by X-ray diffraction. Crushed powder of cadmium oxalate was subjected to X-ray diffraction analysis using with characteristic Cuka (1.5406 A^0) radiation for structural analysis at CRYSTA PEAK SOLUTION LAB, Pune and fig.3 indicated the x-ray powder diffraction pattern of cadmium oxalate crystal. The sample scanned in 2θ value from 10^0 to 70^0. The characteristic peak appears at 13.88 (2θ) for cadmium oxalate crystal. The 2θ, d value, relative intensity, and Millar indices (h k l) of major peak observed in spectra of cadmium oxalate crystal are given in table 2. The result confirmed that all crystal formed is crystalline in the nature having triclinic structure [16-17]. These results are well agreement with the slandered JCPDS data [53-0085]. By using Debye – Sherrer's formula, the grain size calculates as D = 83.60 nm from the XRD data, the lattice parameter was found as a = 6.00 A^0, b = 6.66 A^0, c = 8.49 A^0 i.e. $a \neq b \neq c$, $\alpha = 74.66$, $\beta = 74.28$, $\gamma = 81.00$i.e. $\alpha \neq \beta \neq \gamma$ and unit cell volume V=314.30 A^0. In triclinic crystal structure the length of unit cell is different and $\alpha \neq \beta \neq \gamma \neq 90^0$.

2θ	d A^0	Relative Intensity (cts)	Indices

733

11.160	7.92208	86.8723	(0 0 1)
13.888	6.37163	2902.32	(0 1 0)
15.420	5.74177	1681.98	(1 0 0)
19.798	4.48078	838.566	(0 -1 1)
24.263	3.66530	1282.45	(1 -1 1)
27.920	3.19307	801.848	(0 2 2)
33.768	2.65224	994.015	(2 0 2)
55.916	1.64305	179.326	(1 -3 2)
57.750	1.59515	108.316	(2 -3 1)

Table 2. XRD data of grown cadmium oxalate crystal

4.2 Fourier Transforms Infrared Spectroscopy (FTIR)

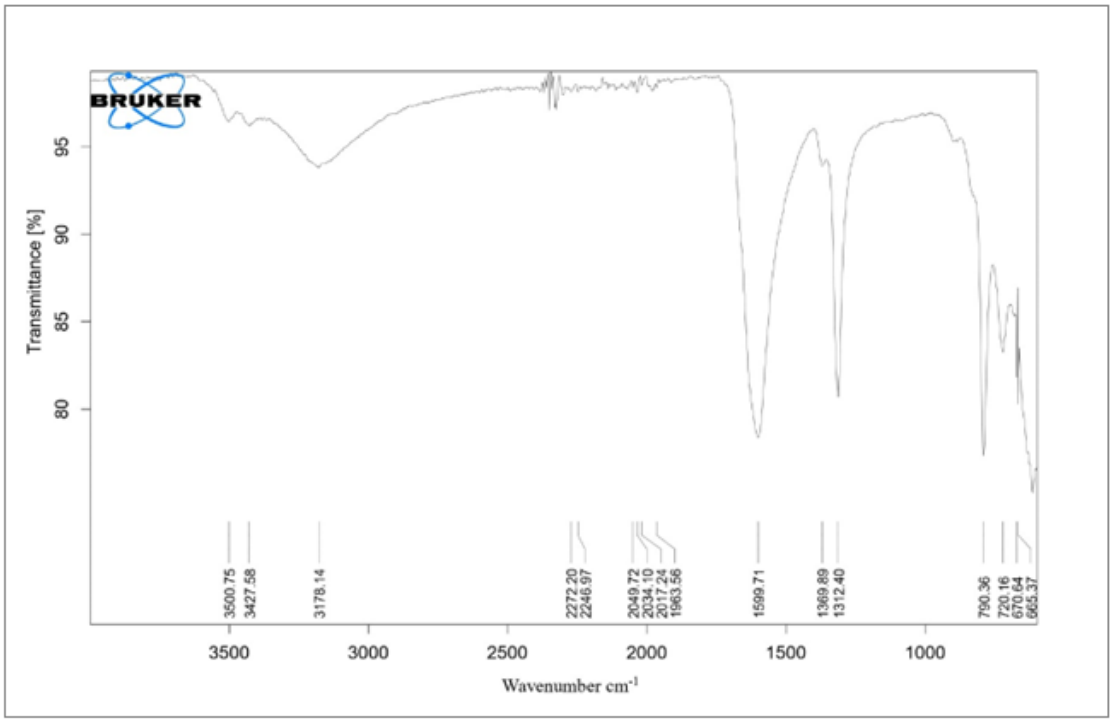

Figure 4. FTIR spectrum of the grown cadmium oxalate crystal

The infrared spectroscopic technique is one of the best powerful tools for investigating functional group in compound [18-19]. The functional group present in grown crystal had been analysed using Fourier Transforms Infrared (FTIR) spectrum. The FTIR spectra of cadmium oxalate crystals were recorded using KBr pellet technique in the region 4000 cm^{-1} to 400 cm^{-1} using Shimadzu FTIR – 8400S spectrometer at R. C. Patel Pharmacy Research Institute Shirpur. Fig.4 shows the FTIR absorption spectrum of cadmium oxalate crystals grown in agar-agar gel. The fundamental FTIR frequencies observed in grown crystal [20]. The bonds assigned to the different type of vibrations are summarized in table 3. The spectrum shows peak in the region 3500 $cm^{-1}$to 500 cm^{-1}. The bonds in the range 3500cm^{-1} to 2240 cm^{-1} is attributed to symmetric and asymmetric O-H strong stretching of water molecule. The bonds at 1599.71 cm^{-1} recognized to C-C stretching group [21]. The presence of C-O stretching vibration bond at 1369.89 cm^{-1} and 1312.40 cm^{-1}. The C-H bending bond observed at peak 790.36 cm^{-1}. The peak 665.35 cm^{-1} can be attributed metal oxygen bond.

Sr. No	**Wave Number cm^{-1}**	**Assignments**
1.	3427.78, 2272.20, 2246.97	O-H stretching and water molecule

734

2.	1599.71	C-C
3.	1369.89, 1312.40	C-O
4.	790.36	C-H
5.	665.35	Metal oxygen bond

Table 3. FTIR data of grown cadmium oxalate crystal

4.3. Scanning Electron Microscope (SEM)

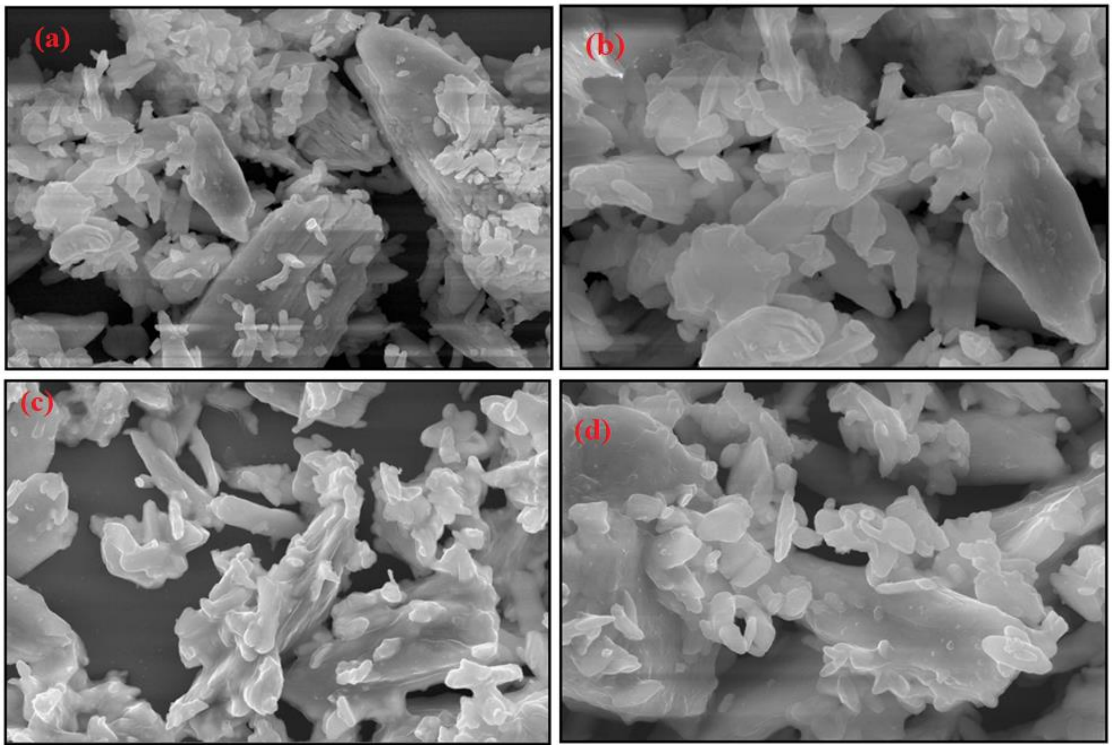

Figure 5. SEM image of the grown cadmium manganese oxalate crystals

Scanning electron microscope (SEM) is used to study of surface of grown crystal. The SEM gives information about internal structure of crystal. It is also used to verify the presence of imperfection. In the present work morphology of powdered sample cadmium oxalate crystals were studied by scanning electron microscope. SEM analysis was carried out at CRYSTA PEAK SOLUTION LAB, Pune. Fig.5 shows various SEM photograph of cadmium oxalate crystal. It was investigated that a coarse mixture of compound of various particle size. SEM images shows that crystal had composed of many sheets approximately greater than 1163 nm in length. This compound is predominantly crystalline, characterized by the formation of various shapes with regular phases. The SEM images show crystal growth by layer deposition with oval, capsulated, cocci, columnar, triangular, rod, spherical, polygonal, elliptical, opal, ruby, garnet, and spades shape.

5. CONCLUSION

1. White colour with average size of 7x3x2 mm^3 single crystal of cadmium oxalate crystals obtained during a period of 3 weeks at room temperature by using single diffusion technique in agar- agar gel.
2. The XRD pattern shows that grown crystals are in crystalline in nature and having triclinic structure.
3. FTIR spectroscopy of cadmium oxalate investigated the presence of oxalate ligands and crystallization water molecule.
4. SEM image suggest that grown crystal has different morphology like oval, capsulated, cocci, columnar, polygonal, elliptical, triangular and rod shapes.

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REFERENCES

1. D. Govindhan, B. Kullaiah, P. Vishwanath, D. Michael, "Handbook of Crystal Growth, Springer Heidelberg Dordercht, New Work, Londan.**2010**.
2. I. V. Markov, "Crystal Growth for Beginners," World Scientific Publication, **2003**.
3. S. J. Nandre, S. J. Shitole, R. R. Ahire, "International Journal of Chemical and Physical Sci," Vol-3, PP.123-130, **2014.**
4. C. R. Ronda, T. Justel, H. Nikol, "Journal of Alloys and Compounds," Vol-275, PP.669-676, **1998.**
5. S. M. Dharmaprakash, P. M. Rao, "Bull Master.Sci, Vol-8, PP. 511-517, **1986.**
6. H. S. Pawar, S. J. Nandre, S. D. Chavahan, R. R. Ahire, "Journal of Emerging Technologies and Innovative Research," Vol-8, PP. 625-634, **2021.**
7. S. K. Arora, T. Abraham, "Journal of Crystal Growth," Vol-52, PP.851-857,**1981.**
8. A. Pactor, "Kristall and Technik," Vol-12, PP.729-735, **1957.**
9. J. Dennis, H. K. Henisch, "Journal of Electrochemical Society," Vol-114, PP. 263-266, **1967**.
10. M. M. Sanjeevannanavar, N. Jagannatha, P. S. Rohith, "AIP Conference proceeding," Vol-2220, PP.6009(1)-6009(5), **2019.**
11. P. S. Rohith, N. Jagannatha, K. V. Pradeep, K. L. Susheela, "Journal of Application Chemistry," Vol-8, PP. 1838- 1844, **2019**.
12. M. R. Shedam, A. R. Venkateswara, "Master.Chem.Phys," Vol-52, PP.263-266, **1998**.
13. M. R. Shedam, R. M. Shedam, S. N. Mathad, "Journal of Nano-ElecronPhys," Vol-8, PP. 4075(1) – 4075(4), **2016**.
14. D. Arumugan, A. Krishan, "Journal of Nanomaterials and Biostructures, Vol-8, PP. 1835- 1843, **2013**.
15. K. M. Chauhan, S. K. Arrora, "Crystal Research and Technology, Vol-44, PP.189-196, **2009**.
16. A. M. Raj, D. D. Jayanthi, V. B. Jothy, "Solid State Science," Vol-10, PP. 557-562, **2008**.
17. V. Mathivanan, "Physis Thesis," Karunya University, Dept. Physis, **2013**.
18. N. B. Colthup, L. H. Daly, S. E. Wiberley, "Introduction to Infrared and Raman Spectroscopy," Academic Press, New York, USA,**1990**.
19. A. K. Patil, U. S. Jagtap, H. R. Talale, "International Journal of Basic and Applied Science," Vol-11, PP.108-113, **2022.**
20. Y. R. Sharma, "Elementary Organic Spectroscopy," S.Chand publication, **2008.**
21. P. V. Dalal, "Indian Journal of Material Science," Vol-10, PP. 01-05, Article 682950, **2013.**