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GROWTH AND THERMAL CHARACTERIZATION STUDY OF CADMIUM OXALATE CRYSTAL IN AGAR Gel

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Abstract: Synthesis of cadmium oxalate crystal grown by gel method using agar- agar gel media, at room temperature. In the present investigation, the cadmium oxalate single crystal was grown by single diffusion techniques. By using different parameter like aging period, concentration of reactants, percentage of gel, reversing of reactants found that the effect on growth of cadmium oxalate was studied. Such good quality crystals were found in quadrilateral, platy, cubic as well as diamond shape. Thermal behavior of material studied by using thermogravimetry (TG) and differential thermal analysis (DTA). It is investigated that the material is thermally stable up to 46^0 C beyond which it decomposes through many stages till the formation of cadmium oxide at 780^0C.Elemental analysis studied by energy dispersive Analysis by X-rays (EDAX) and confirmed that presence of cadmium in sample.

Keywords - Gel Grown Cadmium Oxalate Crystal, TGA/DTA, EDAX

I. Introduction

Crystal growth has become an important part of material science and engineering. Good quality and good size crystal is needed for preliminary testing information as well as production of good quality device. The single crystal is one of the most importance group of material because single crystal having uniform, continues and highly order structure. Single crystal gives authority to possess unique properties thus unique nature can allow transmission without scattering of electromagnetic wave. The progress in crystal growth was highly demanded in view of its recent advance in the field of amplifier, transducer, semiconductor, ferrites, infra rays, non-liner optics, piezoelectric, photosensitive material, microelectronic and microprocessor [1]. Crystal growth in gel is superior and simple technique for growing single crystal for metal oxalate [2] and transition metal oxalate [3] due to their solubility in water [4]. In the different field transition metal oxalate is important having many applications. By using silica gel many research grown crystal [5-9] and very few research grown crystal using the agar- agar gel method [10-11]. The gel technique is simple, low cost as well as not pH dependant. Crystal can grow at room temperature so many research must select gel method for crystal growth. The work on cadmium oxalate is reported only silica gel and rarely [12 -13] in literature attempt on the growth of cadmium oxalate crystals using agar agar gel method. cadmium oxalate crystal is an extraordinary compound has wide range of application. Therefore, the purpose of the present work is to report growth and influence of various parameter on the growth mechanism of cadmium oxalate in agar gel at room temperature. We have reported in this paper to grow and discuss the effect of various parameter on cadmium oxalate crystal growth using agar-agar gel method. In the present course cadmium oxalate crystal growth using agar -agar gel by using single diffusion technique.

II. EXPERIMENTAL WORK

In the present research work, to synthesis cadmium oxalate crystal grown by using agar-agar gel method. Cadmium Chloride ($CdCl_2$), Oxalic acid ($H_2C_2O_4$) and agar- agar powder were used as chemical compound. All the chemical compounds were AR grade. The all the experimental solutions were prepared using distilled water. A single class tube having length 15 cm and diameter 2.5cm and 250ml glass beaker were used as crystalizing vessel. In single diffusion, the test tube was filled by first reactant cadmium chloride of desired volume (2ml - 15ml) and molarity (0.2M – 2.5M). Agar –agar gel prepared by mixing (0.5g – 2.5g) agar powder in 100ml double distilled water at boiling temperature, hot agar-agar gel was poured in the test tube and kept 24-48 hours for setting. After setting and aging the gel the second reactant which oxalic acid solution (0.5M -2.5M) of desired volume and molarity was added over the set gel. The open end of tube was closed with cotton plugs and kept undisturbed at normal temperature. After 5-6 days some nucleation was seen on the interface of test tube, then different shape of shine crystals observed in the test tube was separated from the gel washed carefully with acetone and dried. As the grown crystal were collected and observed. The reaction which command to the growth of cadmium oxalate crystal were expressed as,

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

III. RESULTS AND DISCUSSION

Optimal control parameter leading to good cadmium oxalate crystal growth are obtained. The optimum condition for growing good cadmium oxalate crystals is shown in table 1. Different parameter such as concentration of first and second reactant, percentage of gel, aging period has significantly affected the growth rate. The aging period changes while other parameter kept constant, it was found that rate of

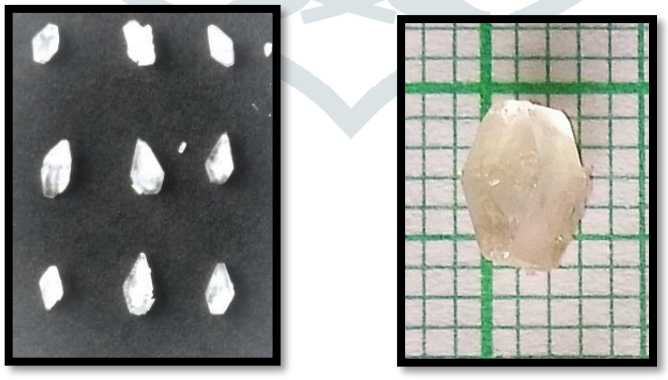
Table 1. Optimum condition for growth of cadmium oxalate

Condition	Single diffusion
Concentration of cadmium chloride	1M
Concentration of oxalic acid	1M
Percentage of gel	1%
Volume of cadmium chloride	5ml
Volume of oxalic acid	5ml
Gel aging period	24 hours
Period of growth	28 days
Temperature	Room temperature
Quality	White, transparent, Quadrilateral, kite, platy and diamond shape

growth was fast at 24 hours. When percentage of gel increased 1% to 2.5%, size and shape of number of crystals were decreases. Fig 1(a), (b), (c) show effect on growth of cadmium oxalate crystal in different concentration of cadmium chloride. It was observed that, as the amount of concentration of cadmium chloride (0.5M to 1.5M) was increased, the size shape and number of crystals were increased. Good quality of grown single crystal having quadrilateral, platy and diamond shaped were obtained as shown in fig 2. The effect of aging period on the growth of cadmium oxalate crystal was to be observed, investigated that at 24 hours' crystal growth rate was better than other. There does not seen to be any change in the quality and shape of grown crystals due to use of reversing reactants.

(a) (b) (c)

Fig. 1. Effect on growth of cadmium oxalate crystals in different concentration of cadmium chloride (a) Growth in 0.5M (b) Growth in 1.0M (c) Growth in 1.5M



(a) (b)

Fig. 2. Grown cadmium oxalate crystal (a) different in morphology (b) good size crystal

IV.CHARACTERIZATION

4.1. Thermal Analysis

Recrystallized alumina sample holder was used and the heating rate was 10^0C/min. Thermograms were recorded in the temperature range from 30^0C to 900^0C.The sample was hold for 1.0min at 300^0C to evaporate water due to moisture and then heated from 30^0C to 900^0C at 10^0C/min. The experiment was carried out at national chemical laboratory under a flow rate of 60ml/min.in nitrogen atmosphere. cadmium oxalate crystal in powdered form was taken as sample for thermal studied. Weight of sample was 25.831mg for TGA/DTA.

4.1.1Tthermogravimetry Analysis (TGA) –

TGA curve for cadmium oxalate crystal is shown in fig.3.The percentage of weight loss in different stages of decomposition of cadmium oxalate are presented in table 2. There is concordance between the observed and calculated weight losses. TGA curve shows that compound is stable up to 46^0C. 23.21% loss in weight may be due to dehydration of water molecule in the temperature range 67^0C to 240^0 C. There is no further weight loss up to 260^0 C and hence cadmium oxalate is completely dehydrated.38.83% weight loss in temperature range 263^0 C to 482^0 C from the dehydrated compound corresponds to loss of CO. The weight loss in temperature range 505^0 C to 780^0 C is 16.98% indicating loss of CO_2.The residue remains stable from 780^0 C and decompose the material. is 16.98% indicating loss of CO_2.The residue remains stable from 780^0 C and decompose the material. is 16.98% indicating loss of CO_2.The residue remains stable from 780^0 C and decompose the material. is 16.98% indicating loss of CO_2.The residue remains stable from 780^0 C and decompose the material. is 16.98% indicating loss of CO_2.The residue remains stable from 780^0 C and decompose the material. is 16.98% indicating loss of CO_2.The residue remains stable from 780^0 C and decompose the material [14-15].

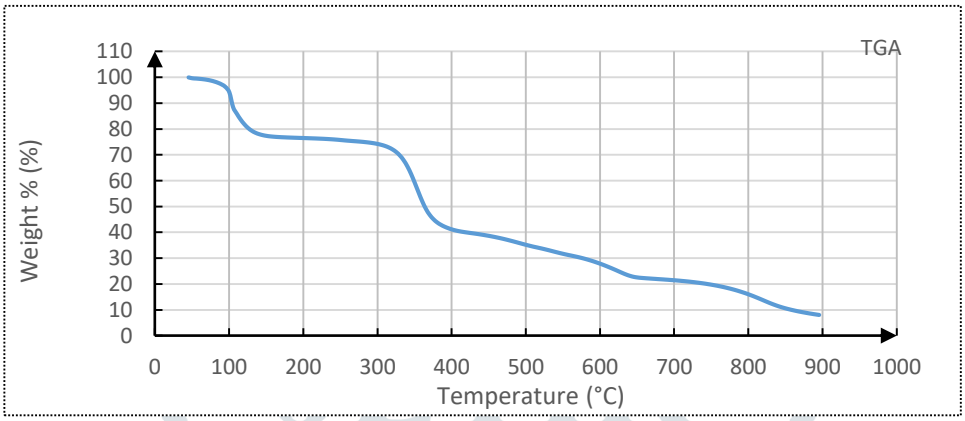


Fig. 3. TGA curve of cadmium oxalate

Table 2. TGA data of cadmium oxalate

Stage	Temperature Range	Observed % Wt. loss	Calculate % Wt. loss	Loss of Molecule
1	67 °C - 240°C	23.21%	23.28%	H_2O
2	263°C - 482°C	38.83%	38.66%	Co
3	505°C - 780°C	16.98%	16.96%	Co_2

4.1.2 Differential Thermal Analysis (DTA) –

DTA Curve for gel grown cadmium oxalate crystal is shown in fig.2 and DTA data collected from this curve is tabulated in the table 3.DTA Curve in nitrogen confirmed the existence of three endothermic peak [2] at 109^0 C, 370^0 C, 620^0 C. In the first stage at 109^0 C, an endothermic peak observed due to loss bulk of water of crystallization. Another endothermic peak at 370^0 C which attributed to loss of water molecule again. The decomposition of oxalate is observed at the onset due to completes dehydration. At 620^0 C endothermic peak represent the decomposition of cadmium oxide into cadmium oxalate [16]. Endothermic peak studied in DTA curves corresponds to total weight loss of water molecule in TG curve.

Table 3. DTA data of cadmium oxalate

Stage	Peak Record	Peak Height	Nature	ΔH (J/g)
1	109.31°C	-65.08%	Endothermic	-65.08
2	369.24°C	-25.60%	Endothermic	-25.6
3	619.75°C	-3.37%	Endothermic	-3.37

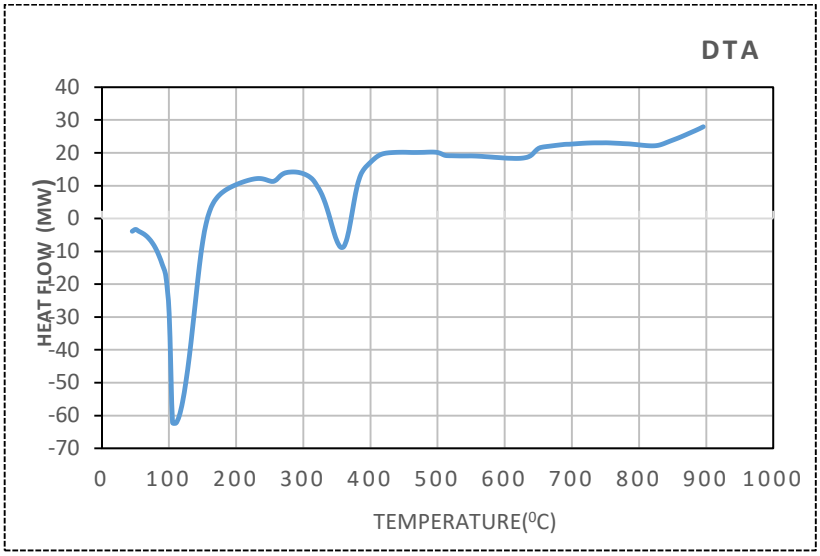


Fig. 4. DTA curve of cadmium oxalate

4.2 ENERGY DISPERSIVE ANALYSIS BY X-RAY(EDAX)

Energy Dispersive analysis by X-Ray (EDAX) is employed for the qualitative analysis and called as elemental analysis. Elemental analysis of cadmium oxalate crystal carried out at CRYSTA PEAK SOLUTION LAB, Pune which standard less at 10 eV energy show the results. The peak ranging from 2.9 KeV to 3.3 KeV clearly indicates the presence of cadmium in the sample. The relative concentration of cadmium is observed as 57.19% EDAX reveals that sample crystal show presence of cadmium. The observed elemental analysis data as shown in table 3 and fig 5 show spectrum of cadmium elemental analysis.

Table 4. DTA data of cadmium oxalate

Sr.No	Element	Shell	Wt%	At%
1	O	k	42.81	84.02
2	Cd	L	57.19	15.98

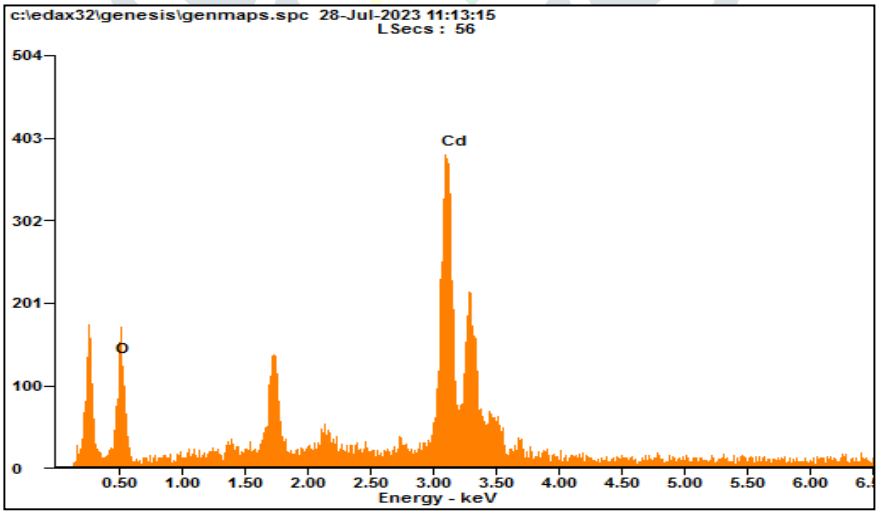


Fig. 5. EDAX spectrum of cadmium oxalate

V.Conclusion

1. Good quality single crystal of cadmium oxalate can be grown by single diffusion method in agar gel method at room temperature.
2. The grown cadmium oxalate crystals were white in colour and having and having different morphology such as quadrilateral, platy, and dimand shaped.
3.Quality of growing crystal is more precise for 1.5M concentration than compare to other.
4.TGA and DTA studied, the water of crystallization present in grown crystal.
5.EDAX Studied suggest that grown crystals are of cadmium oxalate indeed.

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