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Research Article

GROWTH AND CHARACTERIZATION OF CADMIUM OXALATE CRYSTAL USING SILICA GEL

Varsha M. Patil*[1]., Patil H. M[2] and Patil I. J[3]

[1,2]Jijamata Education Society's, Arts, Science and Commerce College, Nandurbar
[3]P.S.G.V.P.M., Arts, Science and Commerce College, Lonkheda, Shahada, District Nandurbar

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ABSTRACT

Single crystals of Cadmium oxalate have been grown in silica gel. The optimum conditions were established by varying the concentration of gel solution and reactants, gel setting time and so forth. Prismatic transparent ring i.e. crystal were obtained. Obtained crystals exhibits triclinic structure with unit cell dimension a =4·3551A°, b = 6·8898A°, c =3·2175A°, α= 90·013A°, β = 97 · 337A°, γ= 101·963A° calculated from x-ray diffractogram. The grown crystals were further characterized with the help of FT-IR studies. Thermal behavior and stability of the grown cadmium oxalate crystals were investigated in the temperature range of 30-1000°c.

INTRODUCTION

Single crystals can be prepared by using the gel grown single diffusion technique. Silica gel method of single diffusion reaction technique is an inexpensive and simple technique for growing single crystals. Crystal growth is heterogeneous chemical process in which conversion from one phase to another phase of compound is involved. Among many methods available, gel technique has become more popular and has been used successfully at room temperature for materials with low solubility including oxalates [1-5]. The purpose of this paper is report the growth and characterization of cadmium oxalate crystal using silica gel.

Experimental details

The growth of Cadmium crystals was carried out in silica gel media by adopting the single diffusio technique. The high purity element such as cadmium chloride, oxalic acid and Sodium metasilicate with AR grade were used as the starting materials in a single diffusion method at a room temperature. The following general chemical reaction was employed for the growth of cadmium oxalate crystals

$CdCl_2 + C_2H_2O_4 \rightarrow Cd\ C_2\ O_4 + 2HCl$

$CdCl_2.H_2O + C_2H_2O_4.2H_2O \rightarrow Cd\ C_2\ O_4 \cdot 3H_2O + 2HCl$

The gel solution was prepared by dissolving Sodium metasilicate powder in double distilled water of specific gravity 1·04 gm/cm^3 with the desired concentrations of Oxalic acid (·5 to 1·5 M) which acted as lower reactant. The gel solutions were transferred to the growth apparatus before setting. Apparatus consist of glass tubes of 20 cm length and 2.5 cm diameter. The pH of gel medium was adjusted between 3·8 to4·5 values. The setting period is varied from 4 to 6 days depending on the setting condition employed.

Feed solution was prepared by dissolving cadmium chloride in double distilled water. Once a gel was set, the feed solution was carefully poured along the walls of the tubes with the help of pipette over the set gel in order to avoid any gel breakage [6].

Experiments were carried out with feed solution of different molarity (0·25 to 1·5 M) and different pH of gel (3·8 to 4·5 pH) to find optimum condition for the growth of crystals. The growth period varied widely in the range of 8-20 days depending on the composition and molarity of feed solution and gel concentration [9]. The optimum conditions are summarized in the table-1.

Table 1 Optimum conditions

pH of the gel	4·2
Concentration of upper reactant	1 M
Concentration of lower reactant	1 M
Gel aging	72 hours
Temperature	Room temperature

Characterization

X-ray diffraction studies of single crystal were carried out by using the intense X-ray wavelength 1·54060A^o. The FT-IR absorption spectrum of as grown Cadmium oxalate crystal was

Corresponding author:* **Varsha M. Patil
Jijamata Education Society's, Arts, Science and Commerce College, Nandurbar

recorded using SHIMADZU FT-IR-8400 spectrometer in the region 400-4000 cm^{-1}. TGA and DTA of grown crystal were carried out to study the thermal stability and decomposition stages of a grown Cadmium oxalate crystal.

RESULTS AND DISCUSSION

XRD Analysis: The XRD pattern of sample is as shown in figure 1. The well-defined peak at specific 2ɵ values shows high crystalline nature of grown crystals [4, 12, 13]. The observed XRD pattern of this work was indexed using the POWD (Interactive Powder Diffraction Data Interpretation and Indexing Program Version 2.2) software package. Calculated lattice parameters and systems of the crystal are shown in table -2.

Table 2 Calculated unit cell parameter

Crystal system: triclinic	
Parameter	**Cadmium oxalate**
a	4·3551A°
b	6·8898 A°
c	3·2175A°
α	90·013A°
β	97 · 337A°
γ	101·963A

Determination of grain size from XRD spectrum:
The grain size is determined by measuring the width of the line with highest intensity peak. The grain size can be calculated by using formula,
Grain size D =0·9/β cos ɵ
Where β full width of half maxima in radian and D is grain size of the crystal.
D= 0·9 X 1·54060/ 0·00366 X Cos (13·57)
= 1·38654/0·00356
= 38·91 nm
The calculated average grain size is 38·91 nm.

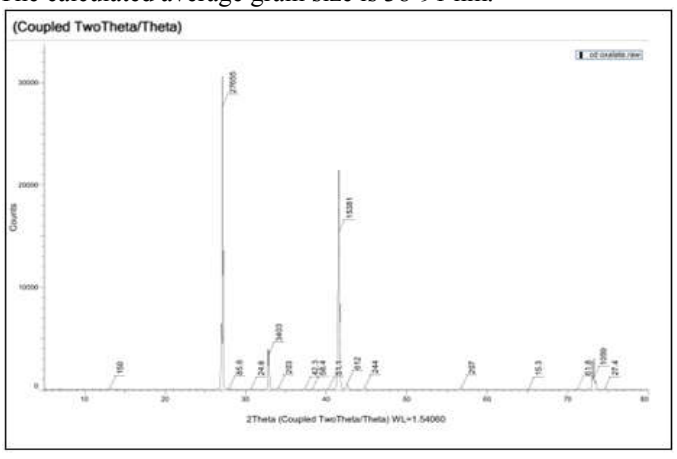

Figure 1 X-ray diffraction pattern of cadmium oxalate

FT-IR Spectrum Analysis

The infrared spectrum in the range of 400-4000 cm^{-1} shows strong band centered at about 3417·98 cm^{-1} which is attributed to O-H stretching vibration of water molecule [11-12]. Vary intense but broad band appeared at 1768·63cm^{-1}which may be due to O-H bending vibration of water molecule [10] and well pronounced sharp peak at 1352·14$cm^{-1}$and peak at 1521·89 cm^{-1}corresponds to the CO_2 symmetric stretching [7]. The detailed band assignments of some selected absorption bands/peaks observed in the FT-IT spectrum is shown in the following table-3.

Table 3 Assignment of some selected FT-IR bands (cm^{-1})

Sr.no.	IR bands (cm^{-1})	Assignment
1.	3417·98	O-H stretching
2.	1718·67	O-H bending
3.	1521·89	Co_2 symmetric stretching
4.	707·90	M-O bond
5.	476·43	Co_2wagging

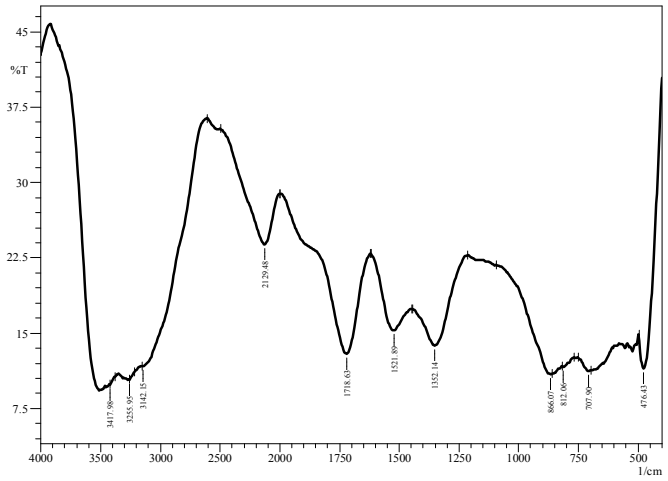

Figure 2 Infrared spectra of cadmium oxalate

TGA/DTA Thermal Analysis

The thermal decomposition of $Cd(C_2O_4)$. $3H_2O$ crystals grown in silica gel recorded in the temperature range of 30-1000^{0}c as shown in fig. Thermogrammetric analysis indicates that the material remains stable up to a temperature of about 75^{0}c. For reaction, It is observed that the first weight loss corresponds to 21.5% of the initial sample weight, which is in accordance with the value derived from reaction (1), there by confirming that the low temperature weight loss could be attributed to the loss of coordinated water corresponding to this dehydration step, there is only one endotherm in DTA at 122.22^{0}c indicating that dehydration takes place in a single step [7,8]. For reaction (2, 3) the percentage mass loss between 300-360^{0}c corresponds to loss of one CO and one CO_2 molecule. Beyond 360^{0}c a stable Cadmium oxide is formed [13].

$CdC_2O_4 \cdot 3H_2O \rightarrow Cd\ C_2\ O_4 + 3H_2O$ ---------- (1)

$CdC_2O_4 \rightarrow Cd\ CO_3 + CO\uparrow$ ---------- (2)

$CdCO_3 \rightarrow CdO + CO_2\uparrow$ ---------- (3)

The DTA peak appearing at 338.48^{0}c shows endothermic reaction which is due to loss of CO_2 and the reaction proceeds to exothermic. The exothermic peak appearing at 350.46^{0}C corresponds to loss of CO. DTA shows clearly that endothermic carbonate formation is followed by exothermic disproportionation [14]. In this temperature range the cadmium carbonate is decompose into cadmium oxide by the release of carbon dioxide.

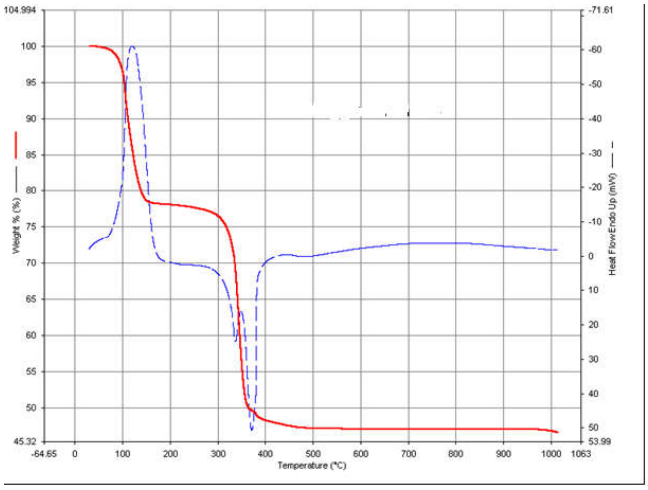

Figure 3 TGA/DTA thermogram as grown cadmium oxalate

CONCLUSION

In view of above experimental observation we may be conclude the following,

1. The growth of single crystal of cadmium oxalate was carried out in silica gel by single diffusion technique. Gel technique is found to be suitable for growth of cadmium oxalate crystal.
2. X-ray diffraction analysis reveals that grown cadmium oxalate crystals exhibits triclinic structure with unit cell dimensions. a = 4·3551A°, b = 6·8898 A°, c = 3·2175 A°, α = 90·013 A°, β = 97 · 337A°, γ = 101·963 A°.
3. The oxalate phase formation was identified from the recorded FT-IR spectra. The FT-IR spectra confirm the presence of coordinated water molecule.
4. From TG/DTA measurements, water of hydration and the chemical formula have been deduced.

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