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Elemental analysis of gel grown Ca-Cd tartrate

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ABSTRACT

Mixed crystals of Calcium-Cadmium Tartrate have been grown by gel technique. The optimum conditions were established by varying various parameters such as pH of gel solution, gel concentrations, gel setting time, concentration of reactants etc. Crystals having different morphologies and habits were obtained. Transparent, pyramidal shaped like diamonds crystals of Calcium Cadmium tartrate were obtained. Some of them were faint yellowish, milky white, due to fast growth rate attached crystals are obtained; faces are well developed and polished. The crystals grown were characterized by X-ray powder diffractometry and Energy Dispersive Analysis by X-rays (EDAX). The results of these observations are described and discussed.

Keywords: Gel technique, Mixed Calcium Cadmium tartrate Crystals, EDAX

INTRODUCTION

With the In recent years crystal growth in gel medium has attracted the attention of many investigators [1-5]. Most of the tartrate crystals are insoluble in water and decompose before melting. Hence single crystals of such types cannot be grown by either slow evaporation or melt method but can be grown by gel method. A number of tartrate crystals grown by gel method have been reported [6-8]. Crystallization methods usually enable to obtain solids of higher purity. Substance with low solubility in water can be grown by the gel technique. The principle relies on the slow migration of crystal constituents (ions) through an inorganic or organic gel so that a very slow reaction occur formation of sparingly soluble compound. When concentration of this compound exceeds the solubility limits, crystals will be formed, the main function of the gel being to control the flow of reacting ions. The present paper reports the growth and elemental analysis of calcium cadmium tartrate.

MATERIALS AND METHODS

The growth of rare-earth tartrate crystals was achieved by using gel encapsulation technique (9). The Crystallization apparatus for the growth of Calcium Cadmium tartrate Crystal's consist of borosilicate glass tube of length 20cm and diameter 2.5cm placed vertically on plastic stand. . The silica gel was used as a growth media.

Gel was prepared by using tartaric acid, Sodium meta silicate having different pH values. The chemicals used for growth mixed tartrate were $C_4H_6O_6$, $CaCl_{2s,}$ $CdCl_2$, Na_2SiO_3, All chemicals were AR grade.

Different molar masses were tried to determined optimum growth conditions. One of the reactants having different concentrations was incorporated in to the gel. This solution was then transferred to several glass tubes. The test tubes were sealed with cotton to prevent fast evaporation and contamination of the exposed surface of the gel. After

setting of the gel it was left for aging for different periods of time. Other reactants' having different concentrations was then added as supernatant over the set gel. Experiments were carried out by changing different concentrations of the reactants.

The chemical reaction inside the gel can be expressed as

$2C_4H_6O_6 + xCaCl_2 + (1-x)\ CdCl_2 = Ca_xCd_{(1-x)}(C_4H_4O_6)_2.xH_2O + 4\ HCl$

The systematic growth experiments were performed by adding $CaCl_2$, $CdCl_2$ as feed solution of strength varying from 0.2 M to 1.2M over the set gel of pH range 4 to 4.5 and gel density range 1.02 gm/cm^3 to 1.05 gm/cm^3.

Observations:
Different parameters such as concentration of reactants, pH of gel, impurities in the solvent, gel setting time, gel aging time, etc have considerable effect on growth rate. Figure 1(a), (b), illustrates different morphologies of pure Calcium Cadmium tartrate crystals grown under different conditions of growth. The crystals grown are Transparent, pyramidal shaped like diamonds were obtained. Some of them were faint yellowish, milky white due to fast growth rate attached crystals are obtained; faces are well developed and polished.

Figure1 (a) Faint yellowish, Milky white, Transparent, Semitransparent, needle shape Well defined crystals of Calcium Cadmium tartrate.

Figur1 (b) Translucent Pyramidal shaped like diamonds crystals of Calcium Cadmium tartrate.

CHARACTERIZATION:
Calcium Cadmium tartrate crystals were characterized by EDAX.

Energy Dispersive Analysis by X-rays (EDAX):
Energy dispersive analysis by X-rays (EDAX) is used for the quantitative analysis and is also called as elemental analysis. In the present work elemental analysis of gel grown calcium cadmium tartrate crystals was carried out at the National Chemical Laboratory, Pune. Figure2. Show EDAX spectrum of calcium cadmium tartrate. EDAX carried out standard less at 30KeV energy shows the following results, for the given sample of the crystal.

Peak ranging from 3.5KeV to3.7KeV shows 1640CPS. It is strong evidence for the presence of calcium because characteristics peak of calcium appears in the range of 3.5KeV to 4.00KeV.The relative concentration of calcium in this sample is 54.2%

Second peak appears between the range of 3.2KeV to 3.5KeV.for cadmium and also at the range of 22.7KeV to 23.6KeV which is broad peak. Characteristics range for cadmium is 3.0KeV to 3.5KeV and 22.5keV to 23.5KeV. So it clearly shows the presence of cadmium in the given sample of crystals. These two peaks give 310 and 40CPS respectively. The relative concentration of the cadmium is 48.8%.

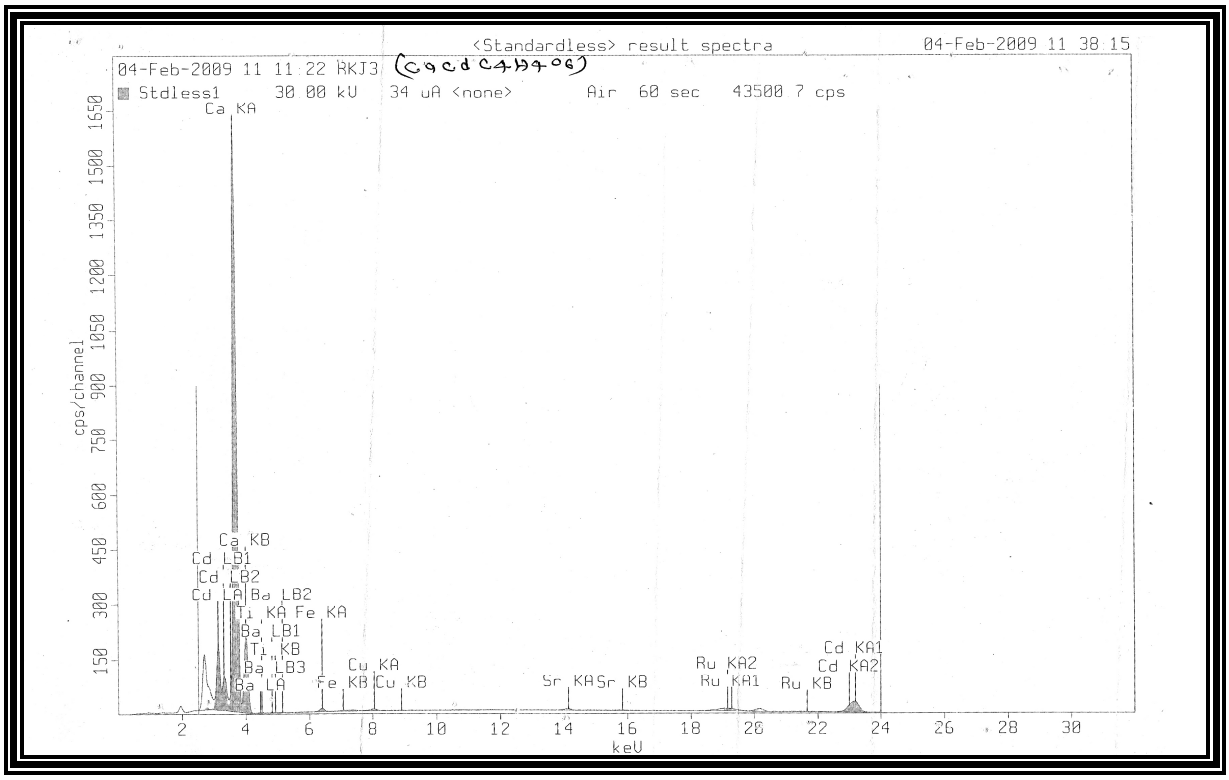

Figure2. EDAX for calcium cadmium tartrate

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