

SHORT COMMUNICATIONS

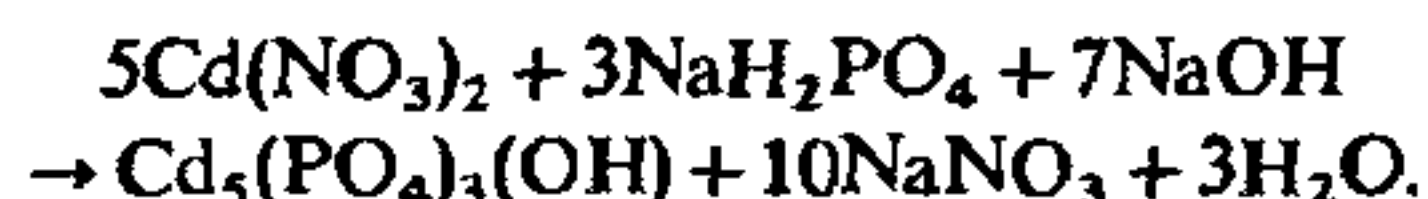
ON CADMIUM HYDROXYAPATITE

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CADMIUM hydroxyapatite, $\text{Cd}_5(\text{PO}_4)_3(\text{OH})$, CdHAp, isomorphous with Hydroxyapatite, $\text{Ca}_5(\text{PO}_4)_3(\text{OH})$, HAp, results when calcium in HAp is completely substituted by cadmium. The present work deals with the synthesis of CdHAp in aqueous media under optimum conditions. The purity of the sample was assessed by chemical analysis, x-ray diffraction, IR, Raman spectra and photomicroscopy. Thermographical (TGA & DTA) analysis of the sample was done to understand the nature of water associated with the sample. Solubility of the sample was investigated under a constant ionic environment of 0.165 M sodium nitrate¹.

Preparation of the sample was based on the following equation:



A solution of cadmium nitrate and sodium dihydrogen phosphate in the stoichiometry desired by the above equation for 10 g yield of the sample were simultaneously added to a 200 ml of CO_2 -free water maintained at pH 9.0 by the addition of a few drops of 0.01 M solution of sodium hydroxide. During precipitation the temperature was maintained at $37^\circ \pm 0.5^\circ\text{C}$ and pH constant by drop wise addition of 0.01 M sodium hydroxide. CO_2 -free nitrogen was continuously bubbled through the medium to keep it well stirred and to eliminate the likely interactions of carbonate during preparation. The precipitate was refluxed with the mother liquor for 6 hr and left overnight to improve crystallinity, filtered, washed well with CO_2 -free water and dried at 110°C for 8 hr. Cadmium and phosphorus in the sample preheated at 800°C for 6 hr was determined complexometrically². (For CdHAp found: Cd 64.06%, P 10.66%; $\text{Cd}_5(\text{PO}_4)_3(\text{OH})$ required: Cd 65.0%, P 10.96%).

The Debye-Scherrer powder diffraction pattern was recorded with a Unicam camera of 9 cm dia using Ni filtered CuK_α radiation ($\lambda = 1.54 \text{ \AA}$) at 35 kV and 20 mA. The crystals of the sample were found to be hexagonal and isomorphous with HAp belonging to $\text{P6}_3/\text{m}$ space group. A comparative study of the x-ray

patterns of the unheated and the sample heated at 800°C showed no change in lattice constants ($a = 9.012 \text{ \AA}$ and $c = 6.614 \text{ \AA}$), intensity and position of lines indicating no new phase was formed on heating. The calculated cell mass, cell volume and crystal density were found to be $2.87 \times 10^{-21} \text{ g}$, $468.18 (\text{ \AA})^3$ and 5.72 g Cm^{-3} respectively. The photomicrograph (figure 1) obtained by a Miopta Binocular Research photomicroscope contained distinct needle-like crystals characteristic of apatites.

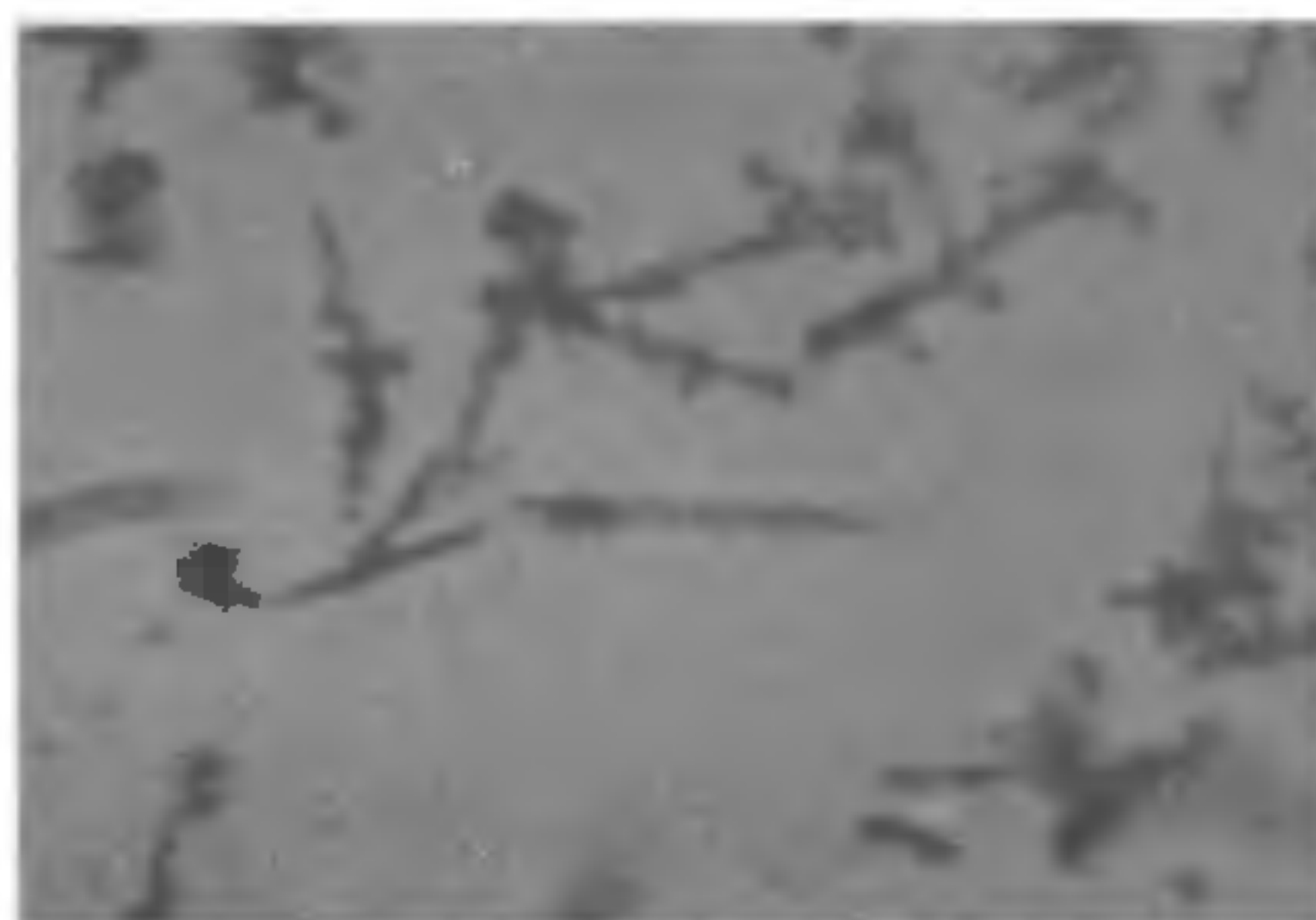


Figure 1. Photomicroscope of cadmium hydroxyapatite ($\times 1000$)

Infrared spectra of the sample was obtained by the use of KBr pellets (0.6 mg sample per 300 mg KBr) with a dry CO_2 -free air purged perkin Elmer 621 IR spectrophotometer in the region $600\text{--}4000 \text{ Cm}^{-1}$. Raman spectrum was taken using plasma filter and scan rate 50 Cm^{-1} . The characteristics IR and Raman absorptions are given in table 1. The observed splitting

Table 1 Assignments of the observed Infrared and Raman frequencies of Cadmium hydroxyapatite.

IR Frequency Cm^{-1}	Assignment	Raman Frequency Cm^{-1}	Assignment
965	$\nu_1 \text{ PO}_4^{3-}$	960	$\nu_1 \text{ PO}_4^{3-}$
1035	$\nu_{3b} \text{ PO}_4^{3-}$	1062	$\nu_3 \text{ PO}_4^{3-}$
1090	$\nu_{3a} \text{ PO}_4^{3-}$	663	$\nu_L \text{ OH}^-$
1660	bending mode of H_2O	3513	$\nu_s \text{ OH}^-$
3538	$\nu_s \text{ OH}^-$		

of the ν_3 band in the IR spectra could be due to more covalent cation-anion bond character in CdHAp.

The thermogram was obtained using a Derivatograph (Hungary) which simultaneously recorded TGA and DTA effects, 27.0% loss in weight was observed in the TG curve at 980°C. Three types of loss of water were seen in the TG curve (figure 2). While loss in weight (TG) between 40°–120°C and a small endo at 105°C (DT) indicated loss of free water, loss between 120°–420°C (TG) and large endo at 322°C (DT) was due to adsorbed; loss between 420°–760°C (TG) and a small endo at 550°C (DT) was due to loss of lattice water. The disappearance of IR absorption peak at 1660 cm^{-1} after heating further indicated the presence of adsorbed water. The activation energy for the process of dehydration was calculated using Freeman and Carroll's equation and was found to be $4.390\text{ kcal mole}^{-1}$.

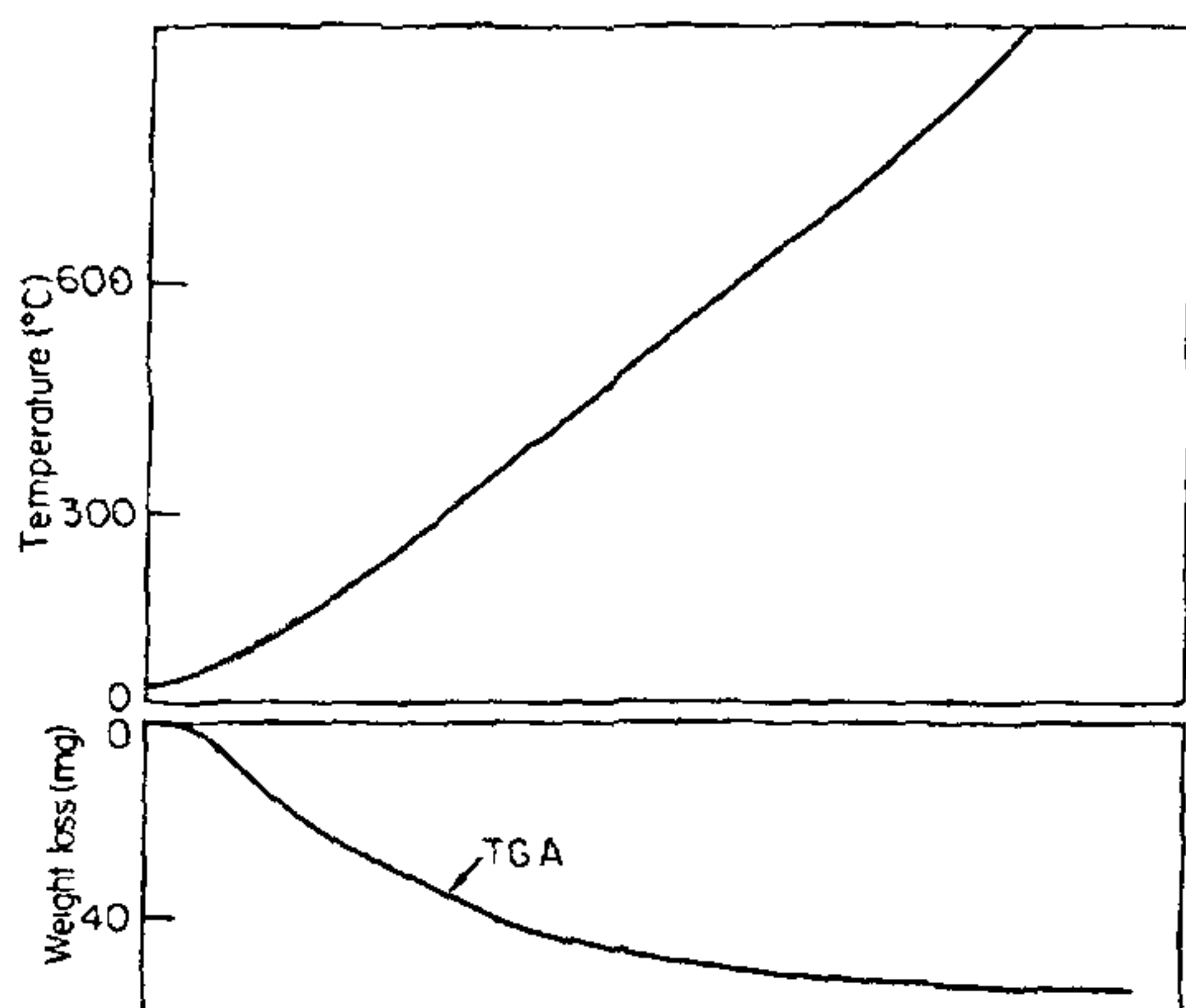


Figure 2. Thermogravimetric (TG) and differential thermal (DT) analysis of cadmium hydroxyapatite.

The solubility of the sample was studied in the pH range 5.0–8.0 at a given temperature and between 37°–52°C at an interval of 5°C at pH 5.0 under a constant ionic environment¹ of NaNO_3 . The solubility was found to decrease with (i) increase of pH and (ii) increase of temperature. The former observation could be explained on the proton accepting tendency of the phosphate ion³. Since CdHPO_4 was established as the virtual solid phase³ controlling the solubility-equilibria of CdHPO_4 at pH 5.0, and since CdHPO_4 too exhibited retrograde solubility⁴, the later observation is thus understandable.

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A BACILLUS SPECIES CAPABLE OF UTILISING DIPICOLINIC ACID AS CARBON AND NITROGEN SOURCE: ISOLATION AND IDENTIFICATION

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THE degradation of dipicolinic acid (DPA) an essential component for true spore formation¹⁻⁴ by the members of *Bacillaceae* has not been studied so far. The present investigation was therefore undertaken to isolate and identify an aerobic spore former capable of utilizing DPA. The appropriate dilutions (1:100) of the soil extract were heated at 80°C for 30 min before inoculating a medium (minerals and DPA). The flask was incubated on a rotary shaker at $30^\circ \pm 1^\circ\text{C}$ and allowed to sporulate. Spores formed were diluted and appropriate dilutions were inoculated into the fresh medium after heating at 80°C for 30 min. This procedure was repeated ten times successively before a single colony was isolated. The media and the methods used for various biochemical tests have been described⁵. The organism was grown in a medium containing (g/l of distilled water) $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, 0.001; $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, 0.01; $\text{ZnSO}_4 \cdot \text{H}_2\text{O} \cdot 7\text{H}_2\text{O}$, 0.01; MnSO_4 , 0.1; $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, 0.2; $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, 0.08; K_2HPO_4 , 0.25 and DPA, 0.4. The solutions of K_2HPO_4 , CaCl_2 and DPA were sterilized separately and added before inoculation. The pH of the medium was adjusted to 7 ± 0.1 before sterilization.