ON CADMIUM HYDROXYAPATITE

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Cadmium hydroxyapatite, Cd₅(PO₄)₃(OH), CdHAp, isomorphous with Hydroxyapatite, Ca₁₀(PO₄)₅(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 nitrate.

Preparation of the sample was based on the following equation:

\[ 5\text{Cd(NO}_3\text{)}_2 + 3\text{NaH}_2\text{PO}_4 + 7\text{NaOH} \rightarrow \text{Cd}_5(\text{PO}_4)_3(\text{OH}) + 10\text{NaNO}_3 + 3\text{H}_2\text{O}. \]

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 CO₂-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° ± 0.5°C and pH constant by drop wise addition of 0.01 M sodium hydroxide. CO₂-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 CO₂-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 complexometrically. (For CdHAp found: Cd 64.06%, P 10.66%; Cd₅(PO₄)₃(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 CuKα radiation (λ = 1.54 Å) at 35 kV and 20 mA. The crystals of the sample were found to be hexagonal and isomorphous with HAp belonging to P6₃/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 Å and c = 6.614 Å), 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 (Å)³ and 5.72 g Cm⁻³ respectively. The photomicrograph (figure 1) obtained by a Microta Binocular Research photomicroscope contained distinct needle-like crystals characteristic of apatites.

![Figure 1. Photomicroscope of cadmium hydroxyapatite (x 1000)](image)

Infrared spectra of the sample was obtained by the use of KBr pellets (0.6 mg sample per 300 mg KBr) with a dry CO₂-free air purged perkin Elmer 621 IR spectrophotometer in the region 600-4000 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>
<thead>
<tr>
<th>IR Frequency Cm⁻¹</th>
<th>Assignment</th>
<th>Raman Frequency Cm⁻¹</th>
<th>Assignment</th>
</tr>
</thead>
<tbody>
<tr>
<td>965</td>
<td>ν₁ PO₄²⁻</td>
<td>960</td>
<td>ν₂ PO₄²⁻</td>
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<tr>
<td>1035</td>
<td>ν₃b PO₄²⁻</td>
<td>1062</td>
<td>ν₃ PO₄²⁻</td>
</tr>
<tr>
<td>1090</td>
<td>ν₃a PO₄²⁻</td>
<td>663</td>
<td>ν₄ OH⁻</td>
</tr>
<tr>
<td>1660</td>
<td>bending mode of H₂O</td>
<td>3513</td>
<td>ν₅ OH⁻</td>
</tr>
<tr>
<td>3538</td>
<td>ν₅ OH⁻</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 1 Assignments of the observed Infrared and Raman frequencies of Cadmium hydroxyapatite.
of the ν₁ band in the IR spectra could be due to more
covalent cation-anion bond character in CdHAp.

The thermogram was obtained using a Derivato-
graph (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 ab-
sorbed water. The activation energy for the process of
dehydration was calculated using Freeman and
Carroll's equation and was found to be 4.390 kcal
mole⁻¹.

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3. Mahapatra, P. P., Mishra, H., Chickarur, N. S.,
4. Mahapatra, P. P., Mishra, H., Chickarur, N. S.,

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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 mem-
bers 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°±1°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 pro-
cedure was repeated ten times successively before a
single colony was isolated. The media and the methods
used for various biochemical tests have been de-
scribed⁵. The organism was grown in a medium con-
taining (g/l of distilled water) FeSO₄·7H₂O, 0.001;
CuSO₄·5H₂O, 0.01; ZnSO₄·H₂O, 0.01; MnSO₄, 0.1; MgSO₄·7H₂O, 0.2; CaCl₂·2H₂O, 0.08;
K₂HPO₄, 0.25 and DPA, 0.4. The solutions of
K₂HPO₄, CaCl₂ and DPA were sterilized separately
and added before inoculation. The pH of the medium
was adjusted to 7 ± 0.1 before sterilization.