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Crystal growth and characterization studies of SMHP single crystal in silica gel medium and laser induced nucleation reduction process

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Abstract

SMHP (strontium magnesium hydrogen phosphate) crystals were grown in silica gel medium in three growth environments using different gel densities, various concentrations of phosphoric acid and supernatant solutions. The gel pH plays an important role in the formation of different HPO_4 species in the phosphoric system. The pH ranges in which HPO_4^{2-} ion dominate were considered, which in turn is necessary for the growth of SMHP crystals. The SMHP crystals are grown in three different growth environments by applying various growth parameters and found the optimum growth environment. The characterization of grown crystals were studied by FTIR, TGA/DTA, SEM, XRD and etching.

Keywords: SMHP; Laser light; Calculi; Surface morphology; Growth parameters; Trace elements and isotopes

1. Introduction

SHP (strontium hydrogen phosphate) and BHP (barium hydrogen phosphate) were grown in silica gel medium at room temperature and reported [1,2]. In present study, an approach is made to grow mixed crystal in silica gel medium at different environments, which contains one major element (phosphate), one minor or trace element (strontium) and one inhibitor (magnesium). SMHP is a mixed crystal, which typically represent the biological crystals formed in the human urinary tracts called renal stones. Strontium (Sr) is a silvery-white alkaline earth metal that exists in several stable radioactive isotopes (e.g. Sr-89, Sr-90). Strontium is the fifteenth most abundant element in nature and it is the most abundant trace element in seawater and thus, it has been become incorporated into all plants and animal tissues. The daily intake of strontium varies from about 1.8 to 2.0 mg/day. Of this, a negligible quantity is supplied by air, approximately 90% by food and the remaining percent by water. Strontium is present naturally in many foods like spices, seafood, cereals, grains and leafy vegetables. There are some evidences that strontium is essential for the growth of animals, especially for the calcification of bone and teeth. Metabolism of strontium closely resembles that of calcium, especially with regard to bone. The absorption of strontium normally varies from 5 to 25% in injected dose. A 70 kg standard man contains nearly 320 mg of strontium. The skeleton contains more than 99% of the strontium. The rest is distributed among soft tissues; the largest concentration resides in the aorta, larynx, trachea and lower gastrointestinal tract. When administrated orally, it is primarily excreted in the faeces. Strontium is excreted in low in the sweat and in the milk of lactating females. When the mineral level of the body fluid increases, automatic mineral deposition starts. Authors have done a series of experiments (in vivo studies) with silica gel as a biological crystal growth medium at different pH values ranging from 5.5 to 10 and have proved that one can obtain the periodic precipitation, Liesegang rings of biological crystals such as HAP, Brushite, Struvite, BMHP, SMHP, etc. [3–5].

2. Materials and methods

The dissociation of phosphoric acid system can be represented by three-dissociation equilibrium and the presence of various ions at various pH values are reported [6]. Based on the account of these results, the gel pH in the range from 6 to 10 has been used (Milwaukee QS-MN pH-600, packet digital pH-meter are used for measurements) in which the HPO_4^{2-} ions

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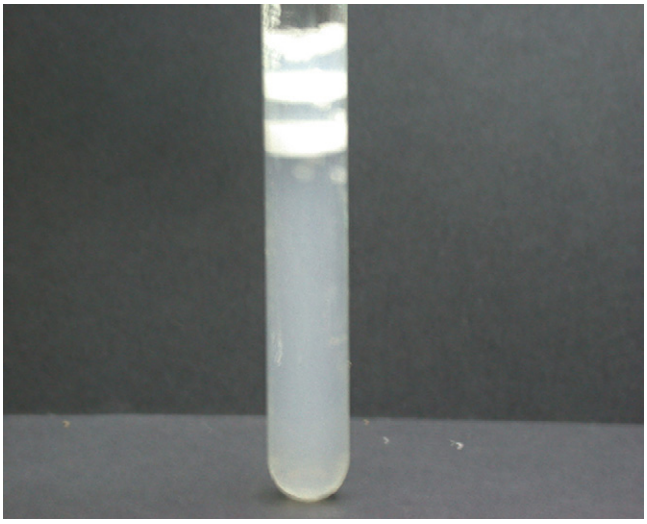

Fig. 1. Growth of SMHP crystals within laboratory environment (SD) at 30 °C (gel density = 1.03 gm/cm^3).

dominates or alone exists. This decreases the possibility of the occurrences of SMP crystals during SMHP growth. The crystallization apparatus employed were glass test tube of 25 mm diameter and 150 mm length for single diffusion method (SD) and thick walled 30 mm diameter and 180 mm long glass U tubes for double diffusion (DD) method. The chemicals used were Excelar-Qualigens (E-Q) AR grade $SrCl_2$ and $Mg(NO_3)_2{\cdot}2H_2O$ (M_W—258.41) and E-Q, AR grade phosphoric acid (Sp.gr.1.75). The SMS gel or water glass was prepared as per the literature [7]. One of the reactant phosphoric acid was mixed with silica gel at desired gel density and elevated temperatures. After the gel set, the supernatant solution (strontium chloride +magnesium nitrate) at a required mole solution was slowly added along the walls of the growth columns (test tubes, U tubes) over the set gels and tightly closed to prevent evaporation. Then the growth systems were allowed to react within the gel medium. The temperatures of the growth columns were maintained by the constant temperature bath continually. The sun light exposed medium, the exposure of sun light in to the SMHP growth medium was only in day time (special glass reflection arrangement in a squire box) that is only 8 h/day and the growth period was 3 months. In these investigations, semiconductor diode Laser were used as a one of the growth environment, the power out put was of 3 mW continuously .The Laser out put wavelength and frequencies were 7000 Å and 4.29×10^{14} Hz. The Laser light was applied to the growth columns from top to bottom by SMPS arrangements.

3. Result and discussion

The SMHP crystals are grown in three different growth faces by applying various growth parameters. Among them, the Laser exposed growth medium shows better nucleation reduction and no crystals were formed, because of the inability to attain super saturation. In sun light exposed medium partial nucleation was observed, since exposure of sun light to the growth medium was only in daytime that is only 8 h/day and the growth period was 3 months (Figs. 1–7, Tables 1 and 2).

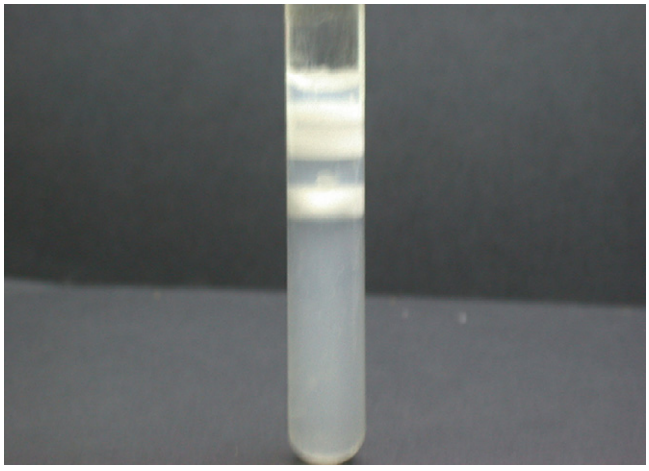

Fig. 2. Growth of SMHP crystals within laboratory environment (SD) at 30 °C (gel density = 1.04 gm/cm^3).

3.1. FTIR spectral analysis of SMHP crystals

FTIR spectrometer having KBr pellets sample holder and KBr detector was used for the analysis. The KBr pellet samples were used to record the spectrum and the absorption frequencies range from 600 to 4000 cm^{-1}. Fig. 8 shows the FTIR spectrum of SMHP crystals. The SMHP FTIR spectrum results matches with Socrater [8]. The absorption bonds, absorption frequencies and percentage of transmittance were recorded and compared with the reported values. The values are tabulated in Table 3. The frequencies values functional groups confirm the SMHP crystal constituents.

3.2. Thermo gravimetric (TGA/DTA) analysis of SMHP crystals

The TGA and DTA of SMHP crystals were carried out by STA 11500-PLTS instruments. The SMHP crystal of 2.439 mg sample was taken for the TGA process. The TGA was started

Fig. 3. Growth of SMHP crystals SD process with the sun light exposed medium at 44 °C.