Growth and Characterization of Magnesium Doped Urinary Crystal in Gel Method

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ABSTRACT   *Urinary stone disease is one of the common diseases in the human history. The prevention of urinary stone formation lies in the exact identification of urinary stone due to the infection of urinary track and urine. Brushite single crystal was grown by gel technique at room temperature by chemical reaction method. Sodium meta silicate ($Na2SiO3.9H2O$) is used as the gel medium.Orthophosphoric acid is used as the inner reactant.Inorder to investigate the growth condition for Brushite crystal in gel at room temperature, magnesium calcium hydrogen phosphate (MCHP) brushite crystals were grown and subjected to various characterizations. Such as powder X-ray diffraction, Fourier transform infra-red spectrum and Ultra-violet visible spectrum analysis. From the powder XRD analysis we confirm crystalline structure. The presence of functional groups was confirmed from FTIR analysis. The UV visible absorption and transmission spectrum of magnesium calcium hydrogen phosphate (MCHP) Brushite crystal were identified and mechanical strength analysed by Vicker's hardness measurement.*

INTRODUCTION

Most kidney stones consist of an organic matrix with bio-minerals. The organic matrix has a composition that remains constant regardless of the type of crystals that make up the stone. The bio-mineral contains hard minerals like Ca, Ba, Sr, Mg and phosphates or its mixtures. The most common and important human body element of all the stones is magnesium [1, 2.3].

There remains a need for controlled, optimization and prospective studies of ionic-substituted brushite materials regarding specific clinical applications, such as, vertebroplasty and kyphoplasty, by studying and defining the needs in terms of cement properties and driving the research towards the adequate solutions. Future developments will enable the commercialization of better and more differentiated products that should improve the clinical outcome and hence the patient life quality[4].

The magnesium calcium hydrogen phosphate (MCHP) also known as Brushite minerals, is a stable form of calcium phosphate crystals were grown in sodium meta silicate in the gel medium. The growth process was done by single diffusion method. MCHP is a compound crystal, which typically represents the biological crystal formed in the human urinary tracts called renal stones. One can obtain the periodic precipitation like Magnesium compounds such as magnesium chloride, magnesium nitrate and magnesium hydroxide that dissolve in water can cause harmful health effect. Magnesium carbonate does not dissolve in water, but does dissolve in the stomach; it can also cause harmful health effects. liesegang rings of biological crystals named as hydroxy Apetite (HAP), Magnesium hydrogen phosphate (MHP) and Magnesium calcium hydrogen Phosphate (MCHP) etc.The MCHP crystal nucleation rate is reduced more in the laser medium that the Sunlight-exposed medium, which is due o variation of super saturation.

EXPERIMENTAL PROCEDURE

Gel is the the majority preferred medium for the in vitro crystallization of biomolecules because of their viscous nature which provide simulation of biological fluids. The gel frame work, which is chemically inert, acts like a three dimensional matrix or a container in which the crystal nuclei are held and supplied with different nutrients for growth. This method is considered as a simplified model to study the crystallization of urinary calculi in vitro [5,6]

To grow the crystal of brushiest in room temperature around 30^{0}C under ambience conditions, the chemicals used were,

Sodium Meta – Silicate ($Na_2SiO_3.9H_2O$), Ortho phosphoric acid (H_3PO_4),Calcium chloride dihydrate ($CaCl_2.2H_2O$) and Magnesium chloride ($MgCl_2$)

25gms of sodium meta silicate was dissolved in 250ml of double distilled deionised water and its density was fixed to 1.03 g/cc and was measured by hydrometer and pH ranging from 6.2 to 7.4 to the gel. The complete dissolved solution was filtered using 0.1 porosity whatmann filter paper. Then above solution is called to Stock Solution [7].

Synthesis:-
The diffusion of Ca^{2+} and Mg^{2+} ions through narrow pores of silica gel leads to the reaction between these ions and HPO_4^{2-} ions of lower reactant already present in the gel suggesting spontaneous nucleation and the expected chemical reaction is (scheme 1):

$$Ca^{2+} + HPO_4^{2-} \longrightarrow CaHPO_4$$
$$Mg^{2+} + CaHPO_4 \longrightarrow MgCaHPO_4$$
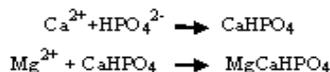
Scheme-1
Crystal growth:-
The mixture of solution had gelled at room temperature, equimolar ratio of (0.5 M each) of Calcium chloride dihydrate and Magnesium chloride were dissolved in deionized water and was added into the upper solution above the gel medium. The upper solution is carefully transferred over in to the gel solution. In order to avoid the damage of gel medium, the upper solution should be added drop wise with the pipette and drops being allowed to fall on the sides of the test tube. The outer reactant of the upper solution is diffuse into the gel medium and reacts with inner reactant.Liesegang rings were observed just below the interface within one (or) two days as shown Figure 1. These rings disappear slowly and tiny platy crystals were observed in it's place. Growth at platy crystals was observed in its place. Growths at platty crystals were observed after to 25 days. The developed crystal as shown in Figure 2. In order to investigate the growth condition for brushite magnesium in gel at room temperature these experiments were repeated by changing growth parameters such as pH ranging from 6.2 to 7.4 to the gel. Solution impregnated with the inner reactants [8].
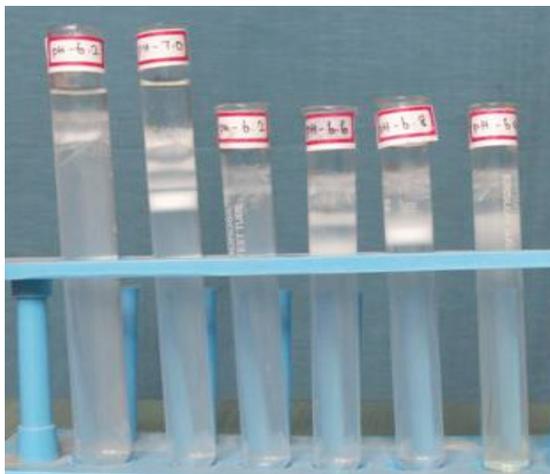
Figure 1: Transparent growing crystals of MCHP in gel

Figure 2: As grown Transparent MCHP crystal

III. RESULTS AND DISCUSSION
Powder X-ray diffraction of MCHP crystal:-
The XRD results revealed that the crystalline property of crystal. The XRD pattern and diffraction indices of the MCHP crystals are recorded (Figure 3) .Using CuK$_\alpha$ radiation of wavelength 1.54 A^0 is the source. Scintillation counter is used as detector. Using powder X-ray diffraction the crystal lattice parameters like a, b, c values were obtained.

a = 6.363A⁰; b = 15.19A⁰ ; c = 5.815A⁰

$\alpha = \gamma = 90°$, β ¹ $118.5°$ a ¹ b ¹ c, $\alpha = \gamma = 90°$ ¹ β

From the above lattice parameters (a, b, c) and angles (α,β,γ) we confirm that MCHP Brushite crystal belong to Monoclinic system.
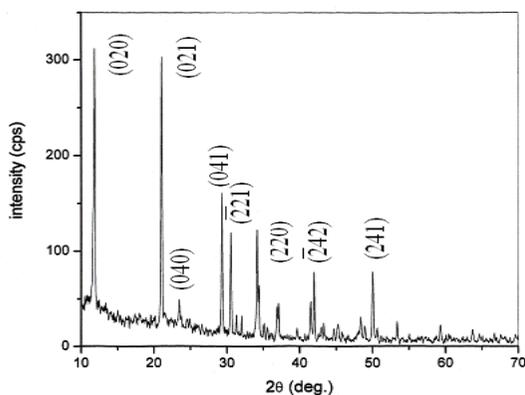

Figure 3: XRD Spectrum of MCHP crystals

FTIR spectral analysis of MCHP crystal:
FTIR spectrometer having KBr pellet sample holder and detector is used for the analysis. The KBr pellet samples are used and the absorption frequencies range from 4000-400cm^{-1}. Figure 4 slows the FTIR spectrum of MCHP crystals. Our results match with the reported values which are tabulated.

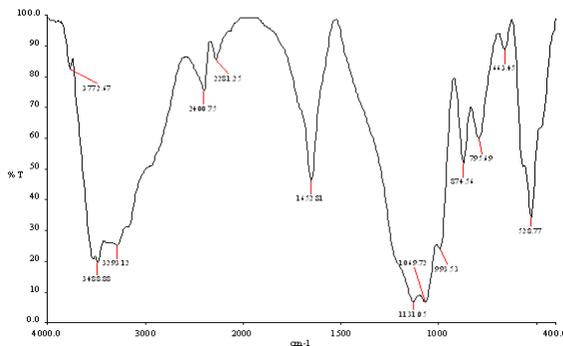**Figure 4: FTIR Spectrum of MCHP crystals**
TABLE -1
.FTIR spectral analysis of MCHP crystal

Reported value Cm^{-1}	Observed value Cm^{-1}	Assignments
3477	3488	Ca – Mg with hydrogen Bond, symmetric (in phase)
1620 -	1652 1131	H – O – H symmetric Stretching bond
987	993	O-O-P asymmetric stretching bond
885	874	C-C bond
525	528	P=O bond

UV Visible Spectral analysis of MCHP Crystal:-
The UV visible spectrum gives information about the structure of the molecule that the absorption of ultraviolet and visible light involves promotion of electrons in σ and Π orbital from the ground state to higher energy state. The UV visible absorption spectrum of MCHP crystal shown in Figure 5.From that spectrum beyond 300nm, the absence of absorption indicates well transparency of crystalline nature.
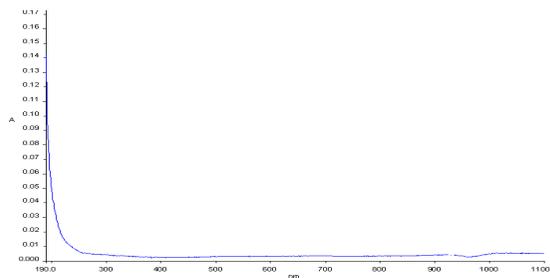

Figure 5:UV-VIS absorption spectrum of MCHP crystals

Mechanical strength analysis of MCHP crystal:-
Microhardness of a crystal is its capacity to refuse to accept indentation. Actually hardness is the resistance to be had by a material to the limited to a small area buckle by scratching or by indentation.The study of mechanical properties of urinary stones difficult and possible only to a limited extent. So, study of the mechanical properties of MCHP crystal will be useful in designing and optimizing the parameters to develop a technique for the urinary stone fragmentation. Vickers hardness indentations are made on the flat face of the crystals (001 plane) at room temperature for loads 25, 50 and 100g using Vickers hardness tester (Figure 6). The average value of the diagonal lengths of the indentation mark for each load is used to calculate the hardness. The crystal is withstand upto 50g and more over it starts crack due to local size indentation effect.
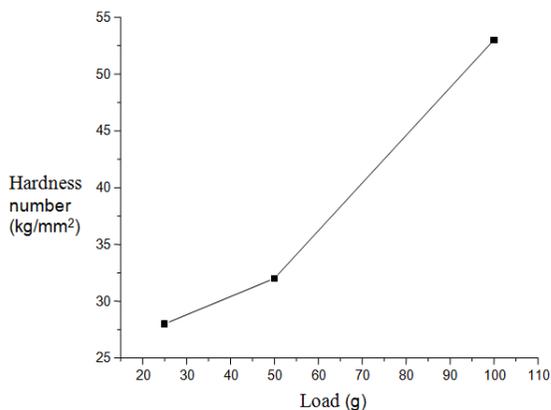
Figure 6: Graph of load versus Hardness number (Hv)

CONCLUSIONS

The MCHP crystal were grown successfully at room temperature .The platy needle crystals were obtained from gel medium of density 1.03 g/cc and pH 6.2 to 7.4 was found to be the best condition for crystallization. The lattice parameters and structure of unit cells are calculated by powder X-Ray diffraction. The XRD data of the crystal confirmed that the MCHP grown crystal system is monoclinic**. The FTIR spectrum of MCHP crystal were recorded at room temperature in the KBr pellet technique and compared with the reported values, which confirm the chemical constituents of MCHP crystals were present. The grown crystals were subjected to UV-Visible spectrum, which shows that the crystal upto 300nm there is no absorption due to crystalline transparency.

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