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Growth of calcium oxalate monohydrate crystal by gel method and its spectroscopic analysis

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Calcium oxalate monohydrate (COM), a urinary stone has been formed by single diffusion gel technique. The grown crystal is analyzed by Fourier Transform Infrared Spectroscopy (FTIR). The spectrum reveals various vibrational modes characterizing particular functional group. The investigation confirms the existence of oxalate group and water molecule in COM .

Keywords: COM crystal growth, Gel method, Functional groups, Crystal growth

1 Introduction

The urinary stones are developed when minerals in the urine clump together and grow instead of being diluted and passed out of the body. They assume various shapes and are composed of many substances. In general oxalate, phosphate, uric acid and urate crystals are found in urinary calculi. Approximately 75% of all stones contain calcium oxalate and additional 5% are composed of calcium phosphate[1]. There are two forms of calcium oxalate; calcium oxalate monohydrate (COM) Whewellite and calcium oxalate dihydrate (COD) Weddelite[2]. The structures of oxalate crystals are shown in Figs 1 and 2. COM exists as elongated rod or sometimes dumbell. COD appears as square envelope. Urinary calcium excretion is influenced by dietary intake of calcium, sodium, protein, carbohydrates, alcohol and potassium[3,4]. The stone formation in our body is similar to the crystal growth[5,6] and can be grown synthetically. The gel method is most versatile and simple technique for growing urinary stones[7]. In the present study, COM is grown by single diffusion gel technique and its functional groups are investigated by FTIR spectroscopy.

Fig. 1—Structure of COM

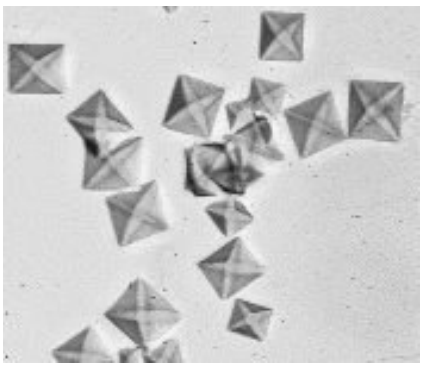

Fig. 2—Structure of COD

2 Experimental Details

Among various gels, sodium meta silicate (water glass) is preferable to grow urinary stones. COM is grown using water glass at room temperature under ordinary conditions. The gel medium provides a polymer grid permitting reactant to diffuse into the

medium with a desirable control rate. The gel solution of required density is prepared from the stock solution.

2.1 Preparation of stock solution

The commercially available water glass of 500 gm is mixed with 1.25 litres of double distilled water. The mixture is then stirred vigorously and continuously to obtain thorough and uniform mixing up of the gel with water. Then the solution is filtered and stored in a container with air tight cork so that the solution may not be affected by oxygen in air and light. This is the stock solution.

2.2 Preparation of gel solution of definite density

The density of the stock solution is determined by specific gravity bottle method. The different quantities of stock solution, i.e. 10, 15, 20 and 30 ml are mixed with double distilled water and the quantities of the final solutions are made equal to 100 ml.

2.3 Growth of COM crystal

Single diffusion technique is employed for the growth of COM crystal. The gel is prepared by treating water glass solution of density 1.03 g/cm^3 with 5% of acetic acid and *p*H is adjusted to 6.35. One of the reactants calcium chloride (1 M) is incorporated inside the gel. After gellation another reactant oxalic acid (0.5 M) is added slowly over the gel. The experiment is conducted at room temperature. A white column of tiny crystals are formed and its functional groups are investigated by FTIR spectroscopy.

3 Results and Discussion

Figure 3 shows the IR spectrum of COM crystal. Brucker IFS 66 V FTIR spectrometer is used to record the IR spectrum of COM crystal. The spectrum shows various frequencies of vibrational modes. Each mode characterizes particular functional group identified from IR correlation chart[8]. The vibrational modes of COM are presented in Table 1.

3.1 Interpretation of vibrational modes of COM crystal

3.1.1 OH stretching

The OH stretching frequency has been used for many years as a test and measure of hydrogen bonds. The stronger the hydrogen bond the longer the OH bond, the lower the vibrational frequency and the broader and more intense the absorption band. For COM crystal OH stretching appears at bands 3500 and 3046 cm^{-1}. Also a band at 3319 cm^{-1} shows intermolecular hydrogen bonded OH stretch[8-11]. This indicated that water molecule is present in COM crystal.

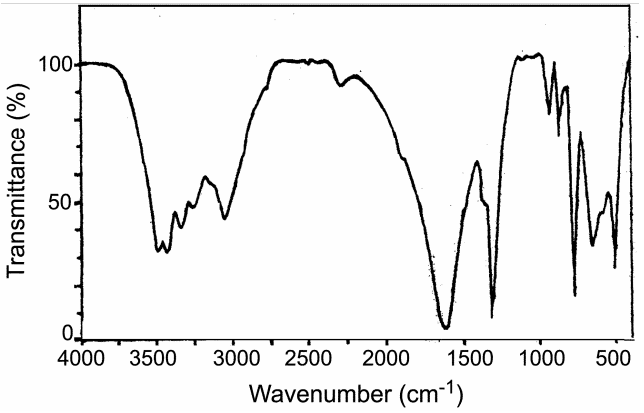

Fig. 3—FTIR Structure of COM

Table 1—Vibrational modes of COM

Wave No. cm^{-1}	Strength	Assignments
3500	S	Asyt.OH stretch
3046	S	Sym. OH stretch
3319	S	Inter mole. Hydrogen bonded OH stretch
1615	V.S	Asym. C=O stretch
1319	V.S	Sym. C=O stretch
1069	V.W	Asym. C-O stretch
1000	V.W	Sym.C-O stretch
865	M	C-C stretch

S – Strong, V.S – Very Strong, V.W – Very weak, M – Medium

3.1.2 C=O and C-O stretching

The C=O stretching absorption in aliphatic compounds generally occurs at 1725-1700 cm^{-1}. The C=O absorption in carboxylic acids appears at 1700 cm^{-1}. If carboxylic acid is converted into its soluble salts, then carboxylate anion is formed. The C=O absorption has possible higher wave number for an acid as compared to that in carboxylate anion. In COM crystal C=O stretching appears at 1615 and 1319 cm^{-1}. The bands at 1069 and 1000 cm^{-1} show C-O stretching[8-11]. This indicates the presence of carboxylate anion in COM crystal.

3.1.3 C-C stretching

The band at 865 cm^{-1} specifies C-C stretching which shows the presence of two carboxylate anion[8-11]. This confirms the existence of oxalate group in COM.

4 Conclusions

COM is grown by gel method. The functional groups of COM are confirmed by FTIR spectroscopy. In vitro growth studies of the COM crystal may give further insights regarding the suppression of the crystal using several inhibitors, which may be useful for pharmacological analysis. Tartaric acid, citric acid, sodium bicarbonate and sodium citrate are some of the inhibitors of urinary crystals[12-14]. These chemicals in small quantities can be added to the gel solution with calcium chloride and their inhibitory action over COM crystal can be investigated. The COM crystal growth requires the excretion of calcium, oxalate and water in the urine. Drinking enough water, a diet low in protein, maintenance of an adequate intake of dietary calcium, restriction of oxalate rich food can prevent COM crystal formation in urine.

References

1 Joseph K C, Bharat B Parekh & Joshi M J, *Curr Sci,* 88 (2005) 1232.
2 Ansari M S, Gupta N P, Hemal A K, *et al.*, *Int J Urol*, 12 (2005) 12.
3 Singh R P, Gaur S S, White D J & Nancollas G H, *J colloid and Interface Sci*, 118 (1987) 379.
4 El Shall H, Jin hwan Jeon, Abdel Aal, *et al.*, *Cryst Res Technol,* 39 (2004) 222.
5 Werness P G, Bergert J H & Simth L H, J *Cryst Growth*, 53 (1981) 166.
6 Khan S R, Whalen P O & Glenton P A, J *Cryst Growth*, 134 (1993) 211.
7 Patel A R & Rao A V, *Bull Mater Sci*, 4 (1982) 527.
8 George Socrates, *Infrared and Raman Characteristic group frequencies Tables & Charts* (John Wiley & Sons Inc.) 3rd edition, 2004, p125.
9 Bouropoulos C, Vagenas N, Klepetsanis P, *et al., Cryst Res Technol*, 39 (2004) 699.
10 Girija E K, Latha S C, Kalkura S N, *et al., Mat Chem Phys,* 52 (1998) 253.
11 Sai Sathish R, Ranjit B, Ganesh K M, *et al.*, *Curr Sci*, 941 (2008) 104.
12 Anusuya A & Sasikala M, *Nutr Res,* 10 (1990) 1109.
13 Meyer J L & Smith L H, *Invest Urol*, 13 (1975) 36.
14 Oyung Jianming, Deng Suiping, Li Xiang ping, *et al., Science in China,* Ser B 47 (2004) 311.