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DISSOLUTION STUDIES IN FEW URINARY TYPE CRYSTALS

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ABSTRACT

Kidney stones are hard, solid particles that form in the urinary tract. The stones were very small and can pass out of the body without any problems. Calcium containing stone, especially Calcium Oxalate Monohydrate, Calcium Oxalate Di- hydrate and basic Calcium Phosphate, Calcium Hydrogen Phosphate Di-hydrate, Struvite and are the most commonly occurring stones. Calcium Oxalate Monohydrate crystals are grown in single diffusion gel growth technique. The grown crystals are characterized by FTIR, XRD and inhibition studies. A number of medicinal plants were antiurolithiatic activity such as Tribulus terrestriis and Boerhaavia diffusa Linn were carried out. Calcium Oxalate Monohydrate crystals in the presence of the herbal extract of one of the medicinal plants Tribulus terrestris Linn. The results are discussed.

KEYWORDS: COM, FTIR, XRD, Medicinal plants.

1.Introduction

Urolithiasiss is a very common and highly recurring painful disease both in develop and developed nations due to the change in life style and food habits(1). In India 15% people are expected to be having urinary stone problem and out of them 50% may lead to loss of kidney or renal damage. Kidney stone is one of the most prevalent diseases and calcium oxalate has been shown to be the main component of the majority of stones formed in the urinary system of the patients. Calcium stones are the most common comprising 75% of all urinary calculi.

Majority of them are COM or (whewellite), COD or (Weddelite), which may be pure of stones of calcium oxalates (50%) and Calcium phosphates (5%) or a mixture of Ca Ox and phosphates (45%). Generally Ca Ox, Phosphates, Uric acid and Urate crystals are found in urinary calculi (2). A number of foods particularly some fruits & vegetables, contain oxalates, notable among these being rhubarb, spinach, sweet potatoes and strawberries. 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 stone is Magnesium. Struvite crystals were grown by single diffusion gel growth technique and the study of the growth inhibition effect on the struvite crystals in the presence of the different concentration of the herbal extract of one of the medicinal plants Boerhaaavia diffusa Linn was carried out (3). In-vitro growth and inhibition studies of citrus limon on uric acid crystals (4).

In the present work Magnesium doped Calcium Oxalate Monohydrate (MgCaOx) crystals are grown by gel technique using single diffusion method. Optimum growth conditions of crystals are determined. Optimum conditions were established by various parameters. The grown crystals are characterized by FTIR, XRD, and SEM-EDX analysis. Incubation of MgCaOx crystal growth was observed in the plant extracts Boerhaavia Diffusa Linn.

2.Materials and Methods

2.1 Crystal Growth

The growth of MgCaOxM crystal was carried out in silica gel. All the chemicals used in this experiment are of AR grade. The borosilicate glass test tubes of 2.5 cm diameter and 20cm length were used as crystallizing vessels. In a single diffusion gel method, gel was set by mixing Sodium Meta Silicate solution of density 1.03g/cm^{-3} was adjusted to a PH of 6 by adding 5% glacial acetic acid (5). Calcium chloride and Magnesium chloride one of the reactants was incorporated inside the gel. After the gel was set an aqueous solution of oxalic acid solution was carefully poured along the walls of the tube with the help of pipette over the set gel, after the gel setting with few days white column of tiny crystals. The growth was completed within a period of 21 days were grown which are as shown in Fig 1. The harvested MgCaOxM crystals are as shown in Fig.2.

Different parameters such as concentration of reactants, PH of gel, impurities in the solvent, gel setting time, etc, have considerable effect on growth rates.

Table. 1. The optimum condition for the growth of MgCaOxM crystals.

S. No	Parameter	Optimum Condition
1	Density of Sodium Meta Silicate	1.03gm/cm^{-3}
2	PH of gel	6
3	Concentration of $CaCl_2$	0.1 Mole
4	Concentration of $CdCl_2$	0.001Mole
5	Concentration of $C_2H_2O_4$	0.1 Mole
6	Gel setting period	2 days
7	Gel aging	1 month
8	Period of growth	21 days
9	Temperature	Room temperature

The reaction between Magnesium Chloride, Calcium Chloride and Oxalic acid in gel medium resulted in the growth of MgCaOxM crystals.

$$CH_3COOH+Na_2SiO_3 \rightarrow 2CH_3COONa\downarrow +SiO+H_2O$$

$$2CH_3COONa+CaCl_2+MgCl_2 \rightarrow (CH_3COO)_4CaMg\downarrow+4NaCl$$

$$(CH_3COO)_4CaMg+C_2H_2O_4 \rightarrow CaMgC_2O_4\downarrow+4CH_3COOH$$

Fig.1. shows growth of MgCaOxM crystal

Fig.2. MgCaOxM crystal

2.2 Characterization Techniques

FTIR spectrum is recorded by KBr pellet technique using Perkin Elmer FTIR spectrometer with the range 400-4000cm^{-1} is available at Centralised Instrumentation Science Laboratory, Department of Physics, St.Joseph College, Tiruchirappalli. Powder X-ray diffraction of the samples are carried out by EXPERT-PRO with CuKα radiation (λ=1.5418A^o) is available at Department of Physics, Alagappa University, Karaikkudi. The morphology of MgCaOxM crystal was studied by SEM and the presence of elemental composition was calculated by EDX method is available at Karunya University, Coimbatore.

3.Results and discussion

The MgCaOxM crystals were grown by single diffusion gel growth technique and the harvested crystals are analysed by FTIR, XRD, SEM-EDX, and incubation studied.

3.1 Fourier Transform Infrared Spectroscopy

The FTIR spectrum of MgCaOxM crystals were as shown in Fig 3. In FTIR spectrum, a strong band at 3433cm^{-1} and 3062cm^{-1} is due to asymmetric and symmetric OH stretching while an intense absorptions band at 3260cm^{-1} show inters molecular hydrogen bonded OH stretch. Intense absorption band at 1621cm^{-1} and 1319cm^{-1} can be assigned to asymmetric and symmetric C=O stretching bands specific to the Calcium Oxalate Monohydrate. The bands at 1094cm^{-1} show asymmetric C-O stretching and 950cm^{-1} shows symmetric C-O stretching. The sharp band at 886cm^{-1} is due to C-C stretching vibrations which confirm the existence of oxalate group in Calcium Oxalate Monohydrate. The sharp peak at 782cm^{-1} is due to O-C=O and the wideband at 665cm^{-1} can be assigned to the bending modes of the water molecule. However, the peak at 517cm^{-1} is assigned to the presence of metal-oxygen bond (6). Thus FTIR reveals that the growth of COM crystals was due to the presence of O-H stretching, C=O, C-C, O-C=O and M=O bonds.

Table. 2. FTIR wave numbers and tentative assignment of MgCaOxM crystals.

Magnesium doped Calcium Oxalate cm^{-1}	Tentative band Assignment
3433	Asymmetric OH stretch
3062	Symmetric OH stretch
3260	Inter molecule H_2 bonded OH stretch
1621	Asymmetric C=O stretch
1319	Symmetric C=O stretch
1094	Asymmetric C-O stretch
950	Symmetric C-O stretch
885	C-C stretch
782	O-C=O stretch
665	OH wagging
517	M-O bond

These peaks are found to be present in the spectrum of MgCaOxM crystals confirms the presence of the element Magnesium.

3.2. Powder XRD Analysis

The powder XRD pattern was recorded using diffractometer system=XPERT-PRO X-ray diffractometer with CuKα radiation (λ =11.546A^0). The powder sample was scanned over the range 10^0 to 60^0 in at a rate of 1^0 per minute.

The powder XRD analysis of the grown COM crystals was matched with the reported database using computer with PAN analytical software and result was matched with JCPDS File (14-0789). The indexed powder data for the MgCaOxM crystals are presented in Table.3. From the collected XRD data, it is observed that from the cell parameters of both pure COM and MgCaOxM crystals belong to monoclinic system.

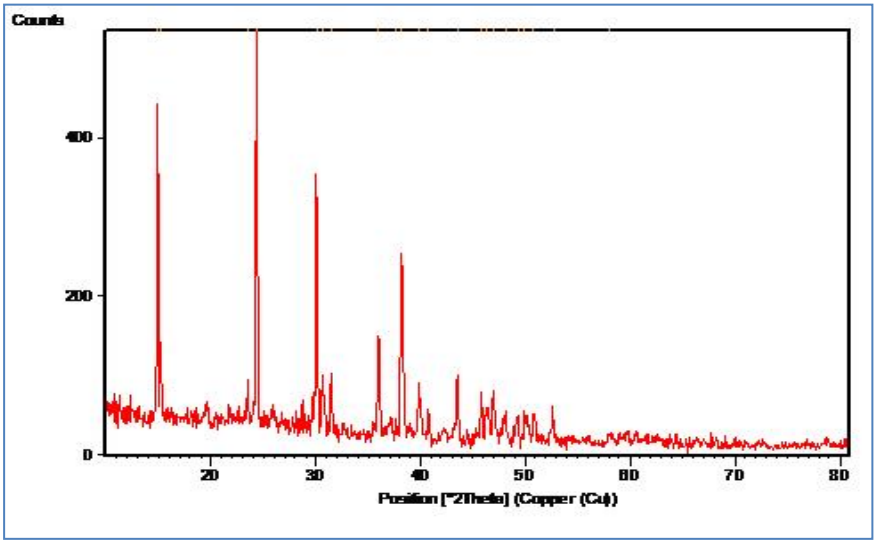

Fig.3. Powder XRD analysis of MgCaOxM crystals.

Table.3. Powder diffraction data of MgCaOx crystal

Observed value		Std value		Observed value	Std value d-spacing	hkl value
2θ	I/I0	2θ	I/I0			
14.864	78	14.927	100	5.95	5.93	101
15.218	23	15.290	60	5.82	5.79	011
23.477	14	23.516	40	3.78	3.77	112
24.356	100	24.366	100	3.65	3.64	020
30.053	56	30.084	80	2.97	2.96	202
30.664	11	30.612	40	2.91	2.91	013
31.436	16	35.965	40	2.84	2.89	121
35.948	24	35.965	60	2.49	2.49	211
37.588	4	37.669	20	2.39	2.38	113
38.1646	43	38.150	60	2.35	2.35	031
39.801	12	39.947	20	2.26	2.26	130
40.732	7	40.796	40	2.21	2.22	204
43.561	15	43.560	60	2.07	2.07	123
45.820	11	45.837	60	1.98	1.99	303
46.3672	6	46.308	10	1.95	1.95	132
46.934	12	46.968	40	1.93	1.93	310
48.1228	6	48.076	40	1.89	1.89	213

The peaks in the XRD patterns which were obtained slightly shifted due to the addition of dopants which indicates that the dopants have entered into the lattice of the crystal. It is seen that the x-ray pattern is almost similar indicating the presence of Magnesium has not affected the crystalline nature of the sample.

3.3 SEM-EDX Analysis

The morphology of the MgCaOxM crystals was studied by SEM. Fig .4.shows the SEM images of MgCaOxM crystals. The grown crystals are obtained in different morphologies such as monoclinic, prismatic, and hexagonal prismatic and plate like shape (7).

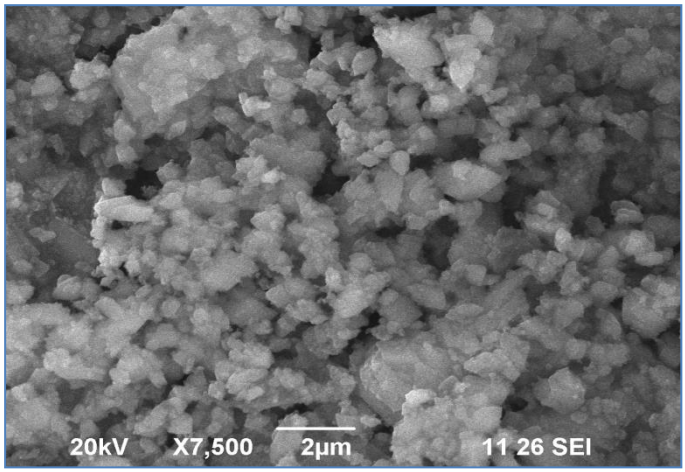

Fig.4. SEM images of MgCaOxM crystals.

It was found that the structure of the grown crystals does not affect the morphology of the crystals by doping.

The presence of Ca and Magnesium quantitative elemental analysis were performed on the application of EDX. The EDX spectrum of MgCaOxM crystal as shown in fig.5.

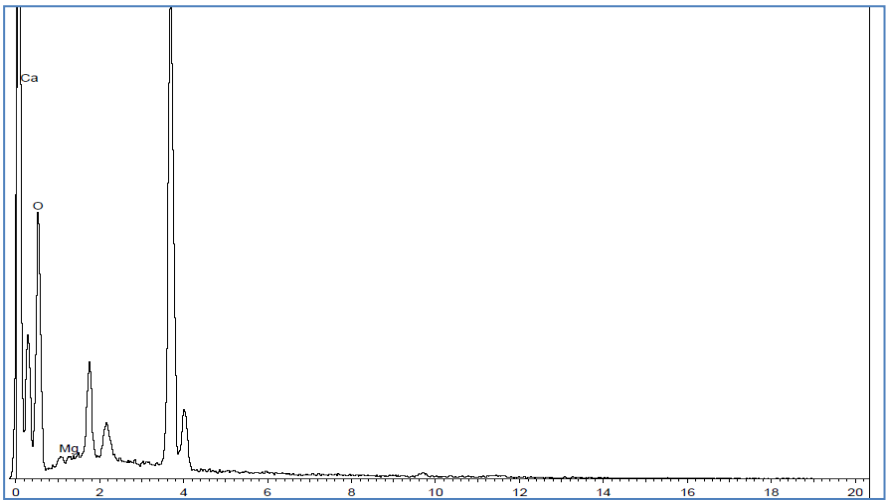

Fig.5. EDX spectrum of MgCaOxM crystals

Table.4. EDX analysis of MgCaOxM crystals.

Element	Mass weight%	Atomic weight%
O	71.25	86.67
Ca	27.61	13.22
Mg	1.14	0.11

The atomic percentage of present element C, Ca, and Mg was found to be 86.67% 13.22 % and 0.11% are present (13).

4.Dissolution of MgCaOxM crystal by plant extract

In the present investigation, the inhibitive study of MgCaOxM crystals is carried out by considering change in the mass value due to the presence of Boerhaavia diffusa Linn extract solution.

The growth and dissolution studies, the MgCaOxM crystals were gently removed from the gel and the tot al mass of the crystals for each concentration was measured.

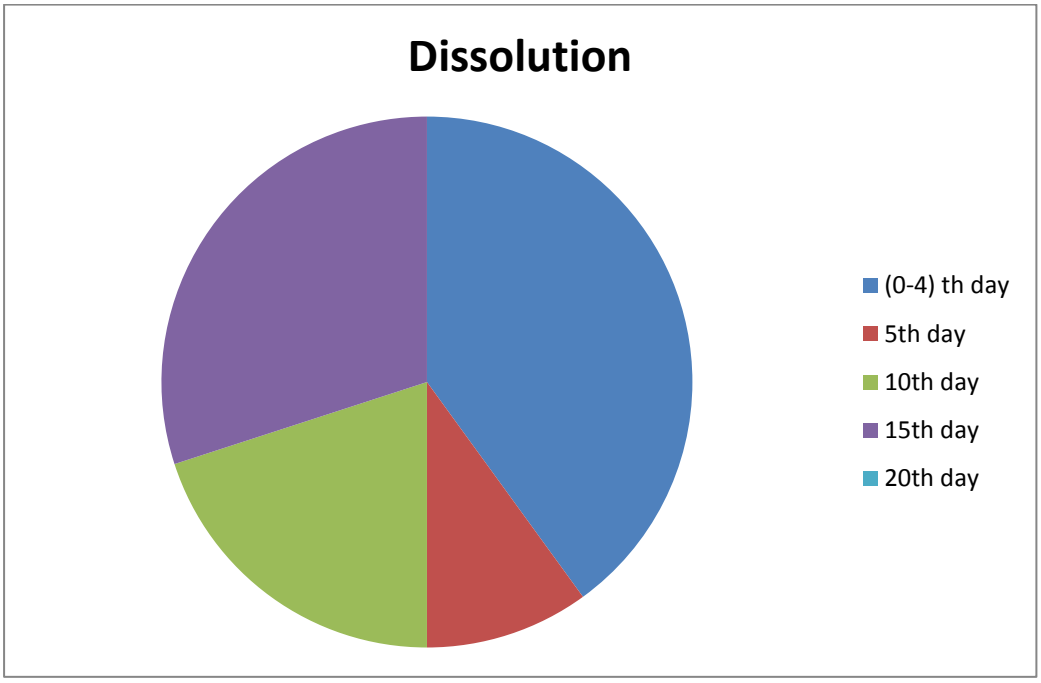

Fig.6. Dissolution of MgCaOxM crystals

From this Fig.6 the Pie diagram represents the total mass on the grown crystals decreased with the increasing concentration of Boerhaavia diffusa Linn. The first- four days of

the crystal was observed by minute change. After 5th day slightly dissolved crystals and less mass weight 75%. After 10th day less dissolved crystals of mass weight 50%. After 15th day moderate dissolved of crystals of mass weight 25%. After 20thday maximum dissolved crystals is zero. This study was useful to MgCaOxM crystals were recurrenced by Tribulus terrestris of urinary calculi formation. The inference is very useful to prevent of urinary type crystals.

5. CONCLUSION

Gel growth technique is used to grown urinary type of crystals. The crystalline structure was identified by XRD analysis. FTIR spectrum of MgCaOxM confirmed the presence of functional groups. The SEM image shows surface morphology of the crystals. The EDX confirmed the MgCaOxM. This study is useful to formulate the necessary dosages to prevent and cure urinary calculi. To increase the acceptability and awareness among the people, there is an urgent need to develop trust and faith towards the safer indigenous system by establishing its validity in treatment for stone diseases. Health care systems are going to become more and more expensive, therefore we have to introduce herbal medicine systems in our health care.

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