Chapter 3

Dielectric studies on struvite urinary crystals, a gateway to the new treatment modality for urolithiasis

3.1 INTRODUCTION

The struvite crystals are biological crystals which are present in the kidney due to the improper renal filtration process. These crystals will aggregate inside the kidney to form a highly recurring painful disease known as Urolithiasis (Ali et al., 2012). The primary cause of the occurrence of struvite crystal is due to the infection produced by the urea splitting bacteria present in the urinary tract (Griffith, 1978; Coe et al., 1992; Hesse et al., 1999). The chemical name of struvite crystal is magnesium ammonium phosphate hexahydrate (MAPH) (Chauhan et al., 2011). The statistical analysis proves that females are more prone to this infectious disease (Joshi et al., 2005). The main treatment modalities existing now for urolithiasis are lithotripsy or surgical removal of stone (Joseph et al., 2005). The surgical removal of stones is highly painful and will be only done in the worst case of disease; hence lithotripsy is the most common and safe treatment modality for urolithiasis (Chauhan et al., 2008). In order to design better lithotripsy treatment parameters, it is necessary to have a detailed in vitro study of the various characteristics of the stones. The biological crystals can be grown in vitro using gel growth technique (Yagyik et al., 1989). This technique will initiate a slow growth of crystals in a gel medium, which act as a matrix. The main advantage of the gel growth technique is that the growth of the crystal can be monitored real time and the conditions of the crystal growth in the test tube imitate the environment inside the body (Singh et al., 1995).

* The work described in this chapter has been published in


The gel growth technique is basically of two different types, namely single diffusion technique and double diffusion technique (Dabhi et al., 2005). The struvite crystals are developed here using single diffusion technique since this provides the simplest in vitro model. The dielectric behaviour of a crystal gives relevant information about the polarizing nature of the crystal (Chauhan et al., 2008; Jun, 2007). The dielectric parameters like dielectric constant, dielectric loss, \( ac \) conductivity, \( ac \) resistivity, impedance, admittance are calculated.

The laser lithotripsy parameters basically depend on the tensile and compression strength of the stones. The present study deals with the new property of the stone, namely dielectric breakdown which provides a gateway for new treatment. In order to overcome the disadvantages produced of laser lithotripsy like high cost of treatment; we are hereby proposing and modelling a new lithotripsy treatment model using the results obtained from the experimental procedures. This treatment modality has been developed with the help of basic dielectric studies of urinary crystals and analysing multiple dielectric parameters. It has been proved by researchers that artificial kidney stones showed some significant dielectric characteristics previously (Ali, 2012).

**3.2 EXPERIMENTAL PROCEDURE**

**3.2.1 GROWTH OF STRUVITE CRYSTALS**

The struvite crystals were grown using single diffusion gel growth technique. Here, the gel was prepared by mixing sodium meta-silicate solution of density 1.03 g cm\(^{-1}\) with 0.5 M of ammonium dihydrogen phosphate solution and the pH was adjusted to 7.2 (Griffith, 1978). The solution was poured into the test tube and kept for gelling for around 48 h. After gelling, 1 M of magnesium acetate solution was added which act as a supernatant solution and it penetrates through the gel and to form struvite crystals as shown in Fig 3.1 (a).

**3.2.2 CHARACTERIZATION OF CRYSTALS**

The optical images of the grown crystals were captured which gives the details of the morphology of the grown crystals as well as the approximate size of the
crystals too. The crystalline nature and crystal type of the developed crystal were confirmed by XRD characterization.

The characterization was done with the instrument PANalytical Model X’pert PRO X-ray diffractometer with Cu-Kα radiation (of wave length 1.54060 Å). The FTIR characterization of the grown crystals was done to confirm the functional groups present in the crystal with the instrument Thermo Nicolet model AVATAR 330 Spectrometer.

3.2.3 PELLET PREPARATION AND DIELECTRIC ANALYSIS

The crystals developed in the test tube were carefully removed, dried and smashed into fine powder. The powdered crystal is made into pellets of thickness $2 \times 10^{-3}$ m and area $1.326 \times 10^{-4}$ m$^2$ by applying a pressure of 9-10 MPa at room temperature (Ashok et al., 2005; Kajal et al., 2007; Navneet et al., 2011; Ansari et al., 2012; Anwar et al., 2013; Aydin et al., 2013; Saikat et al., 2013; Omer et al., 2014) for the dielectric study.

![Fig 3.1](image)

(a) (b)

Fig 3.1 (a) Struvite crystals grown in test tube (b) Optical images of grown crystals.

The dielectric behaviour of the prepared pellet was studied using N4L PSM- 1735 Impedance Analyzer. The temperature was set to vary from 30 °C to 100 °C and
frequency from 1 kHz - 1 MHz. The various dielectric parameters were also calculated.

3.3 RESULTS AND DISCUSSIONS

3.3.1 OPTICAL IMAGE ANALYSIS AND THE CRYSTAL SIZE CALCULATION

The optical image of the developed crystal is captured as shown in Fig 3.1 (b). The image confirms the morphology of the struvite crystal obtained. The crystals are mainly of dendrite, cubical and leaf structure. The surface of the crystals was not so smooth. The approximate size of the crystals can also be measured from the optical images which are about 1.5 cm approximately.

3.3.2 X-RAY DIFFRACTION (XRD) ANALYSIS AND CRYSTAL SIZE CALCULATION

The X-Ray Diffraction pattern of the developed crystal is shown in Fig 3.2. The result obtained was verified with the Joint Committee for Powder Diffraction Standards (JCPDS) data [96-900-7675]. The crystal type and crystalline nature were confirmed to be of struvite. The highest intensity peaks were obtained at 35° and 15° as 2θ values which represent the 100% and 60% peak value for the struvite crystals. The d value 4.257 is the highest intensity peak which is formed in XRD which exactly matches with the standard JCPDS value. These highest peaks are due to the presence of phosphate and magnesium in the developed crystals.

The crystal size is calculated from the peaks obtained in the X-Ray Diffraction pattern by using the Scherrer’s formulae given below (Dinesha et al., 2013):

\[
\tau = \frac{K\lambda}{\beta \cos \theta} \quad (3.1)
\]

where \( \tau \) represents the size of the crystal, \( K \) is the shape factor, \( \lambda \) is the X-ray wavelength, \( \beta \) is the full wave half width maximum, \( \theta \) is the Bragg angle. From the calculation the size of the crystal is obtained as 1.183 cm and the space between the crystal lattice, \( d_{\text{spacing}} \) is 2.79. The crystal size value which is obtained by
calculation and optical imaging comes to be approximately same which proves that the final size of the crystals comes to the range of 1-2 µm.

3.3.3 FTIR ANALYSIS

The FTIR characterization of the sample was done. The Fig 3.3 shows the FTIR spectrum of the sample and this spectrum shows the functional groups present in the sample. The struvite crystal contains magnesium, nitrogen, hydrogen, phosphate groups and oxygen. In the FTIR spectrum shown here, the absorptions at 3415.93 cm\(^{-1}\) and 698.23 cm\(^{-1}\) show the presence of N-H bond, whereas at 3124.68 cm\(^{-1}\) indicates the presence of an O-H bond in the sample.

![Fig 3.2 : The XRD Pattern of the struvite crystal](image-url)
Due to the presence of the above said peaks, it can be confirmed that ammonia and hydrate groups are present in the sample. The absorptions at 1006.84 cm\(^{-1}\) is due to the presence of PO\(_4^{3-}\) ions in sample struvite which confirms the presence of the phosphate group in the sample. The rest of the peaks are due to the carbon bonds present in the sample during its preparation for analysis.

Fig 3.3: The FTIR pattern of the grown struvite crystal

3.3.4 DIELECTRIC RESULT ANALYSIS

3.3.4.1 DIELECTRIC CONSTANT

The prepared pellet was given for dielectric studies. All the dielectric parameters are calculated for a varying temperature starting from 30 °C to 80 °C with a varying frequency from 1 kHz to 1 MHz. The dielectric constant was calculated by using the standard equation.
\[ K = \frac{C_p d}{A \varepsilon_0} \]  

(3.2)

where \( C_p \) is the parallel capacitance, \( d \) and \( A \) are the thickness and the area of the sample, \( \varepsilon_0 \) is the absolute permittivity.

The variation of dielectric constant with applied frequencies is plotted in Fig 3.4 (a). The dielectric constant value will be higher for lower frequencies, whereas it will drastically reduce as the frequency reaches its higher value. In low temperature and low frequency condition, the material will be highly polarized hence the dielectric constant will be high. As the temperature increases the polarizing ability of the material also will be decreased, which shows the drop down of dielectric constant value. The maximum dielectric constant value of the sample obtained here is 175 at 30 °C. The dielectric constant value obtained maximum at 80 °C is 120.

3.3.4.2 DIELECTRIC LOSS

The dielectric loss (Tan D) variation of different frequencies at room temperature is plotted in Fig 3.4 (b). Dielectric loss is a unit less quantity which shows the amount of energy dissipated by the material when it is applied by external fields. As the movement of ions or polarization is high in low temperature and low frequency, the dielectric loss will also be high. The dielectric loss will be gradually decreasing as the frequency increases. The dielectric loss is directly proportional to the dielectric constant value. The maximum dielectric loss obtained here 1.8 at 30 °C. For higher temperature the dielectric loss decreases hence the maximum loss at 80 °C is nearly 0.1.

3.3.4.3 AC RESISTIVITY AND AC CONDUCTIVITY

The \( ac \) resistivity (\( \rho_{ac} \)) and \( ac \) conductivity (\( \sigma_{ac} \)) were calculated using the equations below (Dai et al., 2011)

\[ \rho_{ac} = \frac{2\pi f C t}{A} \]  

(3.3)
\[ \sigma_{ac} = \frac{1}{\rho_{ac}} \]  
\[ \text{.........(3.4)} \]

where \( f \) is the applied frequency, \( C \) is the parallel capacitance, \( t \) and \( A \) are the thickness and area of the sample respectively. The variation in the resistivity and conductivity of the sample for the applied frequencies for different temperature is plotted in Fig 3.5 (a) and (b) respectively.

At the initial stage, for low frequency the ions will start separating which will in turn produce an electric charge flow. Hence at lower frequencies the sample will show high conductivity compared to higher frequencies. Since the resistivity and conductivity are inversely proportional, at lower frequencies the resistance value is almost nullified. As the frequencies increase the resistivity of the crystal will shoot up to 40 \( \mu \Omega \).

3.3.4.4 IMPEDANCE AND ADMITTANCE VARIATION

The impedance and admittance variation for room temperature and higher temperature for different frequencies are plotted in Fig 3.6.

value is inversely proportional to the impedance value (C. Zhou et al.,2008) i.e

\[ |Y| = \frac{1}{|Z|} \]  
\[ \text{.........(3.5)} \]

where \( Y \) is the admittance and \( Z \) is the impedance of the entire set-up. It can be noted that the system impedance and admittance does not vary much for change in the temperature. The variation will depend only upon the change in the applied frequency in the system.
Fig 3.4 : Variation of (a) dielectric constant (b) dielectric loss with applied frequency
Fig 3.5 : Variation of \( a_c \) resistivity (b) \( a_c \) conductivity with applied frequency
Fig 3.6: Variation of (a) impedance (b) admittance with applied frequency
3.4 DIELECTRIC THERAPY FOR UROLITHIASIS

The urinary stones are removed from the body by either surgery or by lithotripsy treatment. The lithotripsy treatment is very common these days which use different external energy to disintegrate the stone inside the body itself without an external cut. The latest among this is laser lithotripsy in which different energy laser pulses will be used to smash the stones, which proves to have a disadvantage in terms of its high treatment cost as well as the current technique proves not to be successful in disintegrating all kinds of stones (Reichenberger, 1988).

The dielectric therapy enters into the new era of lithotripsy treatment. The study proves that there is maximum dielectric loss, in the form of heat, when low frequency signal is applied in the normal body temperature or room temperature i.e., 30 °C. The schematic diagram of the experimental setup is shown in Fig 3.7.

![Fig 3.7: Schematic diagram of dielectric therapy set-up](image)

The mode of giving the external source will be same like laser lithotripsy whereas instead of a laser source, a low frequency signal will be applied through a catheter or fibers which touch the stone (Vivek and Awadhesh, 2011). The low frequency signal of range 100 Hz to 500 Hz signal will be supplied using a pulse generator. The signals will be passed into the body using electropulse probe, which will avoid distortion as well as loss of energy. The probe enters the body and touches the calculi, which will be heated up due to the dielectric heating. As the heat dissipation increases, a plasma state will apply inside the stone which initiates the breakage of the stone (Reynard and Badenoch, 1997). The fibre used to transmit
the signal can be made in such a way that the tip of the fibre can absorb the excess amount of heat without affecting the nearby tissues. The movement of the probe as well as the position and fragmentation of the calculi can be monitored using pyeloscope where it provides real time imaging in the monitor kept outside.

The treatment requires a very simple, low frequency signal generator as the source, which is of very less cost. This meets the disadvantage of the present treatment modality.

3.5 CONCLUSION

The dielectric study of struvite crystals will bring a great breakthrough in the treatment area for urolithiasis. It overcomes all the disadvantages of the existing laser lithotripsy treatment. The stone itself has a property of liberating heat in the form of dielectric loss when a proper external frequency signal is applied, which is proved by the above study. When a low frequency in the range of 500 Hz to 1.5 kHz is applied to the stone it dissipates the heat, even at the normal body temperature which is around 30 °C. This property of the stone can lead to the disintegration of stones which is a gateway to the new design of treatment which can be a breakthrough for the lithotripsy treatment in future.

This study was laboratory based whereas the clinical or preclinical study in association with hospitals was not done due to the lack of dielectric machinery set up in hospitals. The further studies on this treatment modality can be done by taking it to the clinical level with the help of urologists.

The real time implementation of this technique can be done only after testing this working pattern in vivo in patients.