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Structural and Optical Properties of Cetapin XR and Mathumagachooranam Added Struvite Crystals

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Abstract: ***Urolithiasis is a stone disease known from ancient times, but still now the causes for the formation of the different kinds of stones are unknown. The high rate of recurrences of urinary stone disease provides a medical challenge which leads to social and economic problems of considerable magnitude and pursues the researchers to carry out in-vitro studies on these urinary stone crystals. The investigations were carried out to analyse the promotery/inhibitory effects of various allopathic medicines consumed normally for headache, fever, stomach disorder, diabetes etc. The investigations show that the urolithiasis is promoted due to the consumption of allopathic drugs.In this study the Cetapin XR, an allopathic medicine and Mathumagachooranam tablet, are taken for consideration. We have grown three struvitecrystals by adding these drugs separately with 10 mg concentration. The characterization such as powder XRD and UV-Vis spectral analysis are carried out.The crystallite size calculated from the XRD shows that Mathumagachooranam is of small size. The strain values are analyzed using Williamson Hall plot. Due to drug addition the considerable changes have been observed.***
Keywords: Struvite, Gel growth,W-H plot, Strain, Dislocation density.

I. INTRODUCTION

The increasing incidence of crystal deposition diseases such as urinary stones, kidney stones, gallstones, gout etc in people of all ages affecting a considerable number of the total population is a major social and economic problem, considering the number of days lost from work and cost of hospitalization [1, 2]. The increasing level of the body fluid automatically allows the pathological mineral deposition [3]. Supersaturated urine forms the stone in which super-saturation depends on the factors such as urinary pH, ionic strength, solute concentration and complexation etc. It is reported that the possibility of stone recurrence in a person is 67-100% [4, 5] and that mere stone removal cannot provide complete cure for the disease. Drug therapy is suggested to inhibit the growth of existing stone and the formation of new stone. Neither the drug therapy nor the removal of existing stones by physical means can ultimately control the condition of urolithiasis. Hence it is important to understand the mechanism of the stone formation and the identification of the inhibitors and promoters of different crystalline materials present in the urinary calculi. Among the urinary stones, 70 percent are formed by the addition of calcium phosphate and calcium oxalate in various propositions. Apatite and struvite are the two types of Phosphatic stones. The former comprises a series of salts with various calcium/phosphorus ratios, hydroxy-apatite being the commonest. On the other hand, Magnesium ammonium phosphate (MAPH) is a non-calciumcalculi which is generally formed in association with apatite giving rise to the large staghorn calculi which commonly occur in alkaline infected urine. Research explains Calcium oxalate monohydrate (whewellite) nucleation is induced by the drug gentamycin [12]. Also, it has been established that pyridoxine, allopurinol and citrate inhibit the formation of oxalate crystals but the drug allopurinol needs the presence of uric acid to inhibit the formation or else it induces the formation [13 - 15]. A study with urine samples in the laboratory shows that the crystals are easily grown in urine samples obtained from diabetic patients[6]. This gives an idea that drugs used by the sugar patients may promote the stone formation as a side effect. Recently, there has been an increasing interest in growing crystals using gels as a model system for studying crystal deposition diseases in human beings [6-10]. Gel acts as an inert medium during the growth of many crystalline compounds and it acts as an ideal medium in the study of the crystallization of biomolecules in-vitro [11]. The viscous nature of the gel provides simulation of biological fluids in which biomolecules grow. In the present work, an attempt has been made to understand the effect of siddha (Mathumagachooraanam) and allopathic medicines(Cetapin XR) (normally consumed by the diabetic patients) on the growth of struvite crystals. We grow a number of

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struvite crystals in silica gel media with and without adding the above drugs separately on a single concentration of 10 mg to observe the inhibitory/promotery effect of the struvite crystals.

II. EXPERIMENTAL

Glass test tubes of 25mm diameter and 140 mm length are used for the crystallization. All the chemicals used for this technique are AR grade (Merck). Single diffusion gel growth method is employed for growing MAPH crystals. The gel is prepared from sodium meta silicate ($Na_2SiO_3.9H_2O$, molecular weight 284g/mol) and specific gravity of the gel is adjusted to1.03 using deionized water. One of the reactants, 1.5M of ammonium dihydrogen phosphate (ADP), is mixed with sodium metasilicate solution of density 1.03g/cm3, so that the pH of the mixture could be set to 7 by adding 1.5M acetic acid and left undisturbed for 48 hours for the gel to set. After gelation takes place the supernatant solution of 1.5 M of Magnesium acetate ($C_4H_6MgO_4 .4H_2O$} (without medicines) and magnesium acetate prepared with 10 mg of Cetapin XR and Mathumagachooranam are gently poured onto the set gel in various test tubes. After pouring on each supernatant solution, the test tubes were capped with airtight stopples. The experiment is conducted at room temperature. The following reaction is expected to takes place leading to the formation of struvite crystals:

$$NH_4H_2PO_4.2H_2O + CH_3(COO)^2Mg.4H_2O \longrightarrow NH_4MgPO_4.6H_2O + 2CH_3COOH$$

The grown crystals are harvested after 3 weeks by decanting the test tubes and then the gel is removed and washed subsequently with double distilled water and dried to remove the moisture content. The crystals we get are used for further characterization.

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III. CHARACTERIZATION TECHNIQUES

The grown crystals are characterized for structural and optical properties. The structural characterization (Powder XRD) is carried out by XPERTPRO diffractometer. UV-Vis absorption spectra of the grown crystals are recorded using UV-Vis Double Beam Spectrophotometer 2201 in the wavelength range 200-600 nm.

IV. RESULTS AND DISCUSSION

A. Growth

Struvite crystals of different morphologies like needle type and dendritic are grown in the gel. Figure 1, 2 and 3 show the photographs of the grown crystals in gel media.It is observed that the presence of allopathic medicine namely Cetapin XR in the supernatant solution has caused an increase in the number of grown struvite crystals and their average size, whereas Mathumagachooranam tablet added supernatant solution shows a decrease in the crystal size. But while comparing with pure struvite crystal, both the drugs shows decrease in number of grown crystals. The sizes of the crystals are found to be smaller at the bottom of the test tubes due to the formation of the acetic acid at the gel-liquid interface and the diffusion of reactants is comparatively less towards the bottom of the test tubes. The total mass of the grown struvite crystals in each test tube are measured after removal of crystals.The yield of crystals per test tube is obtained for all the samples, from which the promotery or inhibitory effect is understood and is illustrated in Figure 4.



Fig 1: Struvite crystals grown in gel media



Fig 2: Struvite + 10mg Mathumagachooranam



Fig 3: Struvite + 10mg Cetapin XR

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Table 1: Total mass of grown crystals

Medicine and dosage	Total mass of crystals formed (g)
a) Pure struvite	0.9592
b) 10 mg of Mathumagachooranam added struvite (S_1)	0.7445
c) 10 mg of Cetapin XR added struvite (S_2)	0.7622

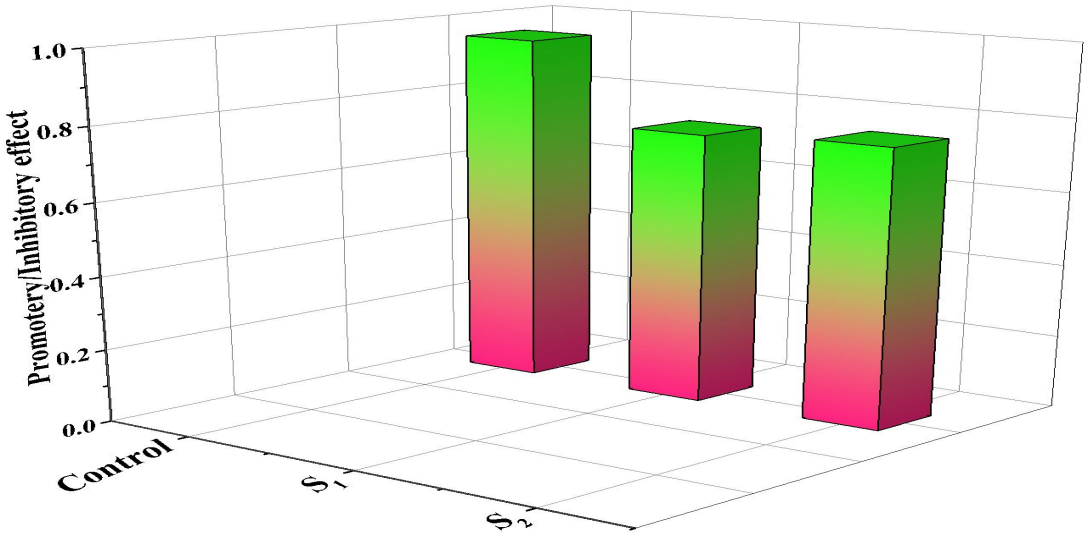


Figure 4: Promotery/Inhibitory effect of struvite crystals

B. Powder X-ray Diffraction

The powder XRD patterns for the undoped and drugs added struvite crystals are shown in the figure 5 and the values of h, k and l in brackets are provided. From the PXRD analysis it is found that the grown struvite crystal crystallizes in the orthorhombic system. The lattice parameters evaluated by PXRD patterns (Table 2) are in good agreement with standard JCPDS (77-2303). The intensity of the diffraction peaks indicates that the samples are well crystallized. The slight shift in reflection peaks, change in intensity of the XRD pattern and change in the values of unit cell parameters and cell volume are noticed in the drugs added crystal and this is due to the incorporation of additional ions into the host of MAPH crystal.

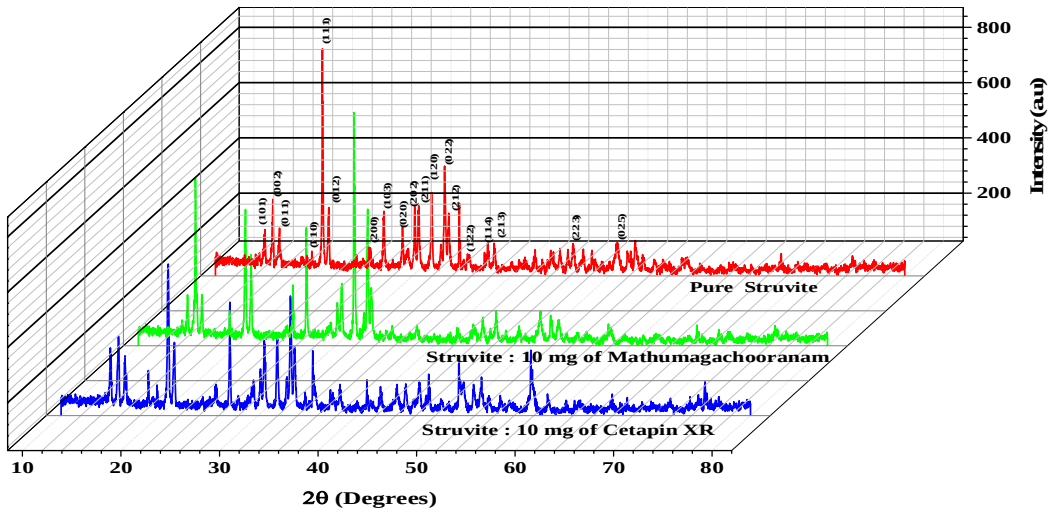


Figure 5: PXRD patterns of undoped and drugs added struvite crystals

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Table 2: The lattice parameters and volume of undoped and drugs added struvite crystals

Samples	Unit cell parameters			Unit cell volume
	a (Å)	b (Å)	c (Å)	V $(Å)^3$
Pure struvite	6.6950	6.1500	11.2058	461.390
10 mg of S_1	6.7429	5.9978	11.2451	454.780
10 mg of S_2	6.9380	6.1935	11.2358	482.807

The crystallite size is determined from the full width at half maximum (FWHM) of the PXRD patterns using the Scherrer formula, $D = K\lambda/\beta cos\theta$,

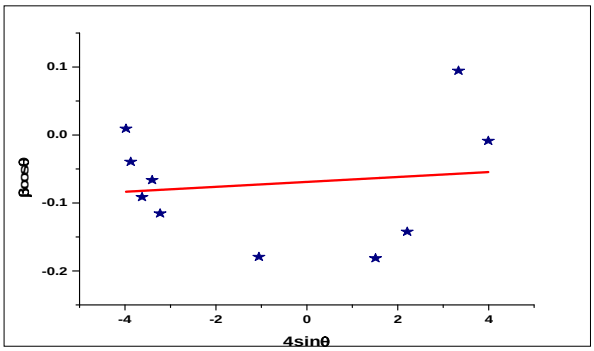
whereD is the crystallite size (nm), K is the constant and usually taken as 0.89, λ is the X-ray wavelength, β is the full width at half maximum value measured in radians and θ is the Bragg diffraction angle. The crystallite sizes estimated using the Scherrer formula are provided in table 3.

C. Williamson-Hall Plot

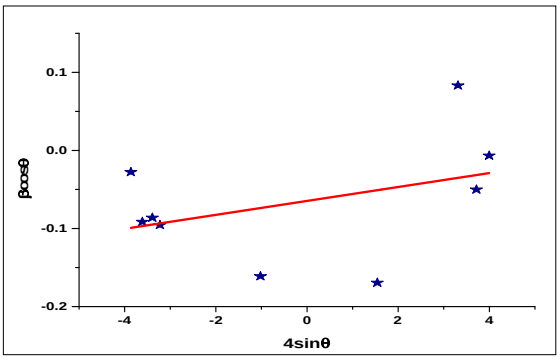
The strain produced by crystal defects are analyzed by using modified Williamson-Hall equation,

$$\beta\cos\theta = \frac{K\lambda}{D} + 4\varepsilon\sin\theta$$

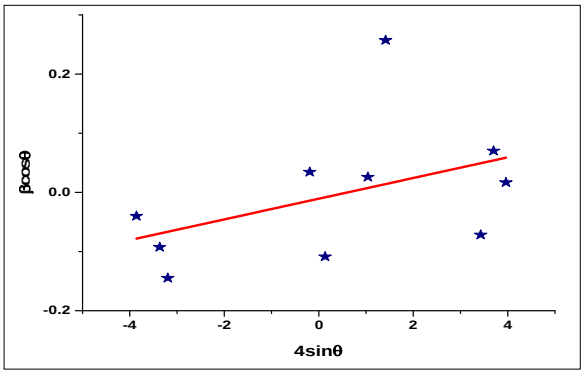
Where λ is the wavelength of X-ray radiation, β is the full width at half maximum, θ is the Bragg diffraction angle, where D is the effective crystallite size, ε is the effective value of micro strain. A plot is drawn with 4sinθ along X-axis and βcosθ along Y-axis. The particle size and strain are calculated using linear fit. The intercept gives the value of D and the slope value gives the value of ε [16].



(a)



(b)



(c)

Figure 6: Williamson plot for (a) pure struvite crystals (b) Mathumagachooranam added struvite crystals (c) Cetapin XR added struvite crystals.

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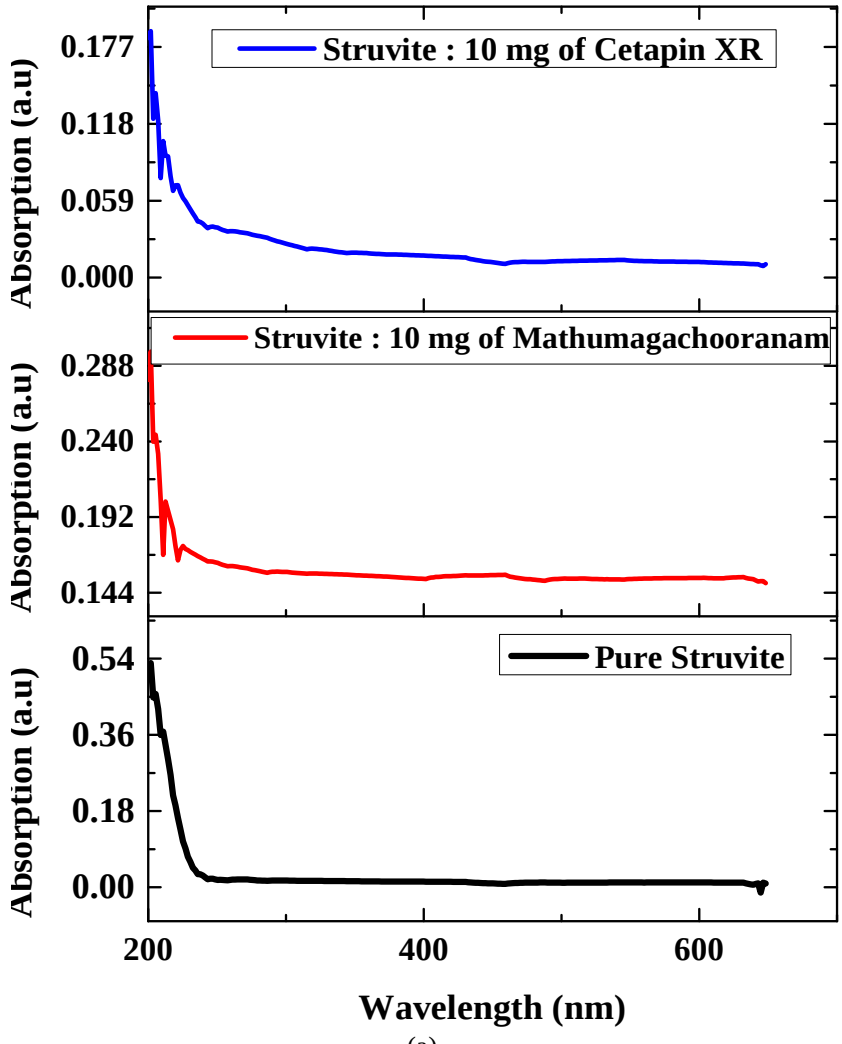
Table 3: Structural parameters of pure and drugs added struvite crystals

Sample	Crystallite size (nm) (Scherrer formula)	Crystallite size (nm) (W-H plot)	The dislocation density($x10^{14}$) (lines/m^2)	Strain ε ($x10^{-3}$)
Pure Struvite	60.56	69.04	2.097	3.62
S_1	56.60	64.66	2.391	8.92
S_2	56.77	72.3	1.913	5.23

Both the drugs change the crystallite size, the dislocation density and the strain compared to those for undoped struvite crystals. The crystallite size for drug Cetapin XR is found to increase (72.3 nm), while the dislocation and strain are found to decrease to 1.913 x 10^{14} (lines/m^2) and 5.23 x 10^{-3} respectively. However, the crystallite size for drug Mathumagachooranam is found to decrease (64.66), while the dislocation and strain are found to increase to 1.913 x 10^{14} (lines/m) and 8.92 x 10^{-3} respectively. The rough decrease in crystallite size, the rough increase in dislocation density and the huge increase in strain occur for the drug Mathumagachooranam added struvite crystals. The changes in the strain may causes the relevant changes in the lattice parameters, accordingly

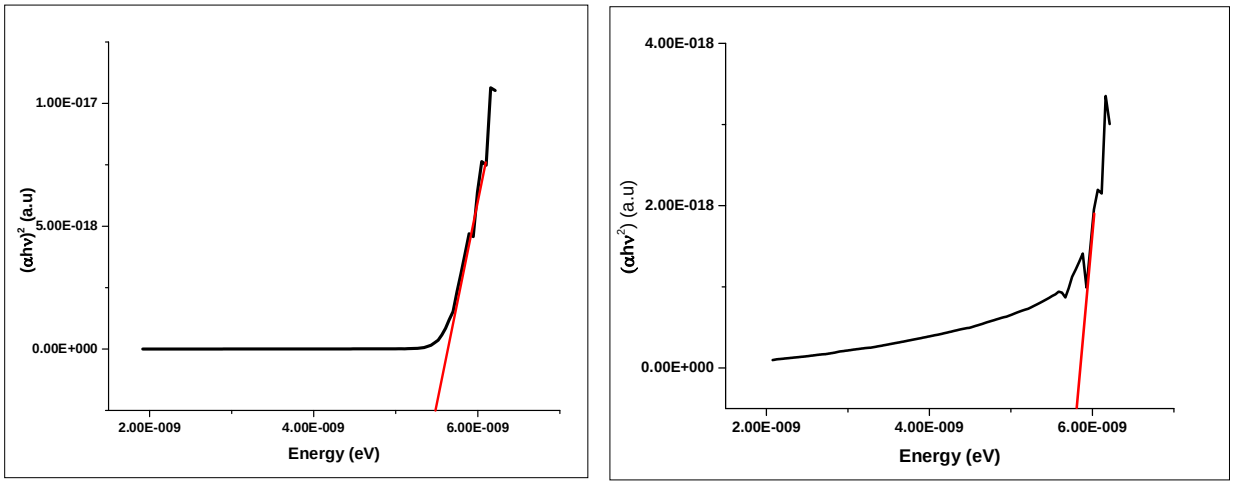
D. UV-Vis Spectral Analysis

The UV-Vis absorption spectra are recorded using UV-Vis Double Beam Spectrophotometer 2201 in the wavelength range 200-600 nm and are shown in the figures 8-10.

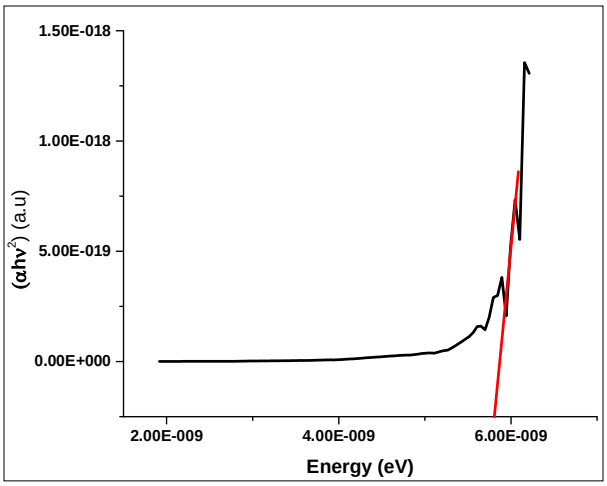


(a)

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(b) (c)



(d)

Figure 7: (a) UV absorption spectra of undoped and drugs added struvite crystals (b) Tauc plot for pure struvite crystal(b) Tauc plot for Mathumagachooranam added struvite crystal (c) Tauc plot for Cetapin XR added struvite crystal.

UV-Vis absorption involves in the excitation of electrons from the valence band to the conduction band. It is clearly seen from the spectra that the samples show good transmission in the entire UV-Vis region. In order to find the band gap energy (E_g), Tauc plot have been investigated by drawing the plot between the incident energy ($h\upsilon$) and $(\alpha h\upsilon)^2$. The band gap energy (E_g), is found by extrapolating the linear part of the plot above the band edge along the incident photon energy ($h\upsilon$) axis where $Eg = h\upsilon$. The band gap for the struvite crystals grown in the control system is found to be 5.48 eV. For Mathumagachooranam and Cetapin XR added struvite crystal, the band gap energy decreases to 5.78eV and 5.80eV.

II. CONCLUSIONS

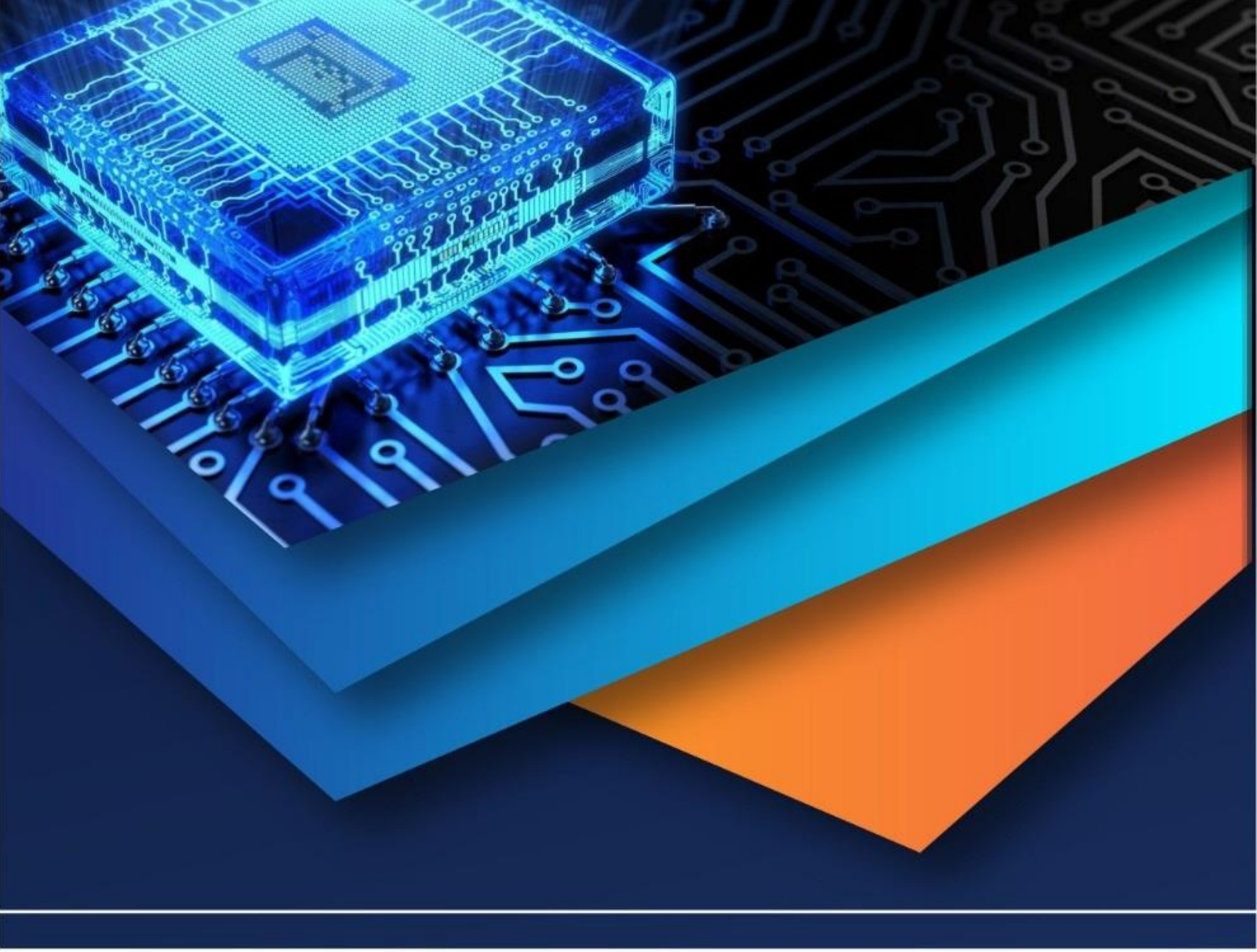
Struvite crystals of different morphologies such as needle and dentritic type are successfully grown using the single diffusion gel growth technique. From the powder XRD studies it is found that the struvite crystals are of orthorhombic structure and reveals the decrease in crystallite size for drugs added struvite crystals. The strain values are calculated from Williamson plot and show an increase for drugs added samples. The UV-Vis analysis shows no significant absorption which means the grown samples are transparent in the entire spectral region. From the present study it is observed that the siddha drug give more inhibition than the allopathic drug thereby indicating the double usage of this drug, one in reducing the sugar and the other in preventing the urinary stone formation.

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