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Growth And Investigations On The Nucleation Kinetics Of Calcium Oxalate Monohydrate Crystal

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Abstract: *The recent research shows most of the urinary stones are grown by Calcium Oxalate Monohydrate (COM). The COM crystal growth requires the excretion of calcium, oxalate and water in the urine. In the present study, COM is grown by solvent evaporation method. The fundamental growth parameters such as solubility, induction period, critical radius, volume free energy change, and critical free energy barrier have been estimated. On the basis of the obtained nucleation data, single crystals were successfully grown.*

Key words: *COM-urinary stone, solvent evaporation, nucleation parameters.*

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. Approximately 75% of all stones contain calcium oxalate [1] and additional 5% are composed of calcium phosphate. The stone formation in our body is similar to the crystal growth and can be grown synthetically [2]. The solvent evaporation method is used in present study which is most versatile and simple technique for growing crystals. The low temperature solution growth technique requires the knowledge of the essential nucleation parameters like solubility, induction period, interfacial energy, volume of free energy, Gibbs free energy and radius of critical nucleus for the growth of good-quality bulk single crystal. Hence, we have investigated the various nucleation parameters of COM for the first time. The grown crystals are confirmed by FTIR and thermal stability analysed by TGA/DTA.

2.Experimental Procedure

2.1 Synthesis Of Calcium Oxalate

Calcium oxalate is a biomaterial with the formula CaC_20_4. This is a white solid, which slowly absorbs moisture to form a white monohydrate. Calcium oxalate powder is dissolved in distilled water completely, to obtain the supersaturate state. The solution was then poured in a clean beaker and covered using aluminum foil with some holes in it for evaporation to takes place. As days go on crystalline takes place and seed crystal keeps on going and attain the maximum size till the solvent get evaporated. The crystal were harvested and good quality (size and shapes is important) crystals are selected for further studies.

2.2 Results And Discussion

Investigations on nucleation thermo dynamical parameters are very essential for the successful growth of good quality single crystals. The solubility and induction period have been determined for the COM solution. Nucleation parameters such as Gibbs free energy, radius of critical nucleus, critical free energy barrier and number of molecules in the critical nucleus and nucleation rate have also been investigated.

3. Solubility Analysis

Solubility factors define the super saturation which is driving force for the rate of crystal growth. Hence for a material to grow as a crystal, determination of its solubility in a particular solvent is an essential criterion. For determining solubility of COM 100 ml of solvent was used throughout experiments. Deionized water is used as solvent COM is less soluble in water, so to enhance solubility a small amount of EDTA was added as catalytic. The solution was stirred continuously, to ensure homogeneous temperature and concentration throughout the entire volume of the solution [3]. After confirming the saturation, the content of the solution was analyzed gravimetrically. The solubility of COM salt was determined at five different temperatures 30,35,40,45 and 50^0 C. The fig .1 shows the variation of solubility with temperature. The graph confirms the solubility diagram has positive slope and so COM crystals can be grown by slow evaporation method [4].

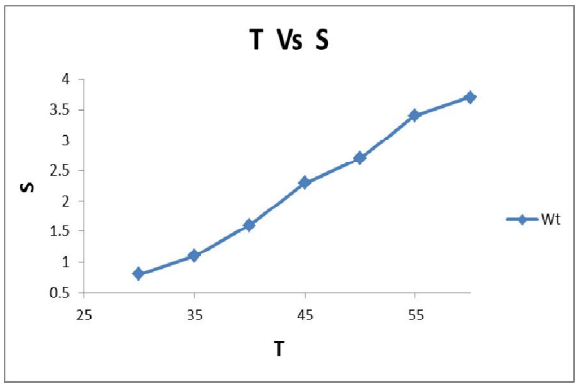

Figure: 1 Solubility curve for COM

4. Determination Of Induction Period

The induction period was recorded the appearance of first visible speck at the bottom of the container and hence the induction period was recorded [5]. The consistency of the reading was verified by repeating the experiment three or four times. Fig 2 shows the variation of induction period with super saturation for COM. It is observed that as the super saturation increases induction period decreases [6].

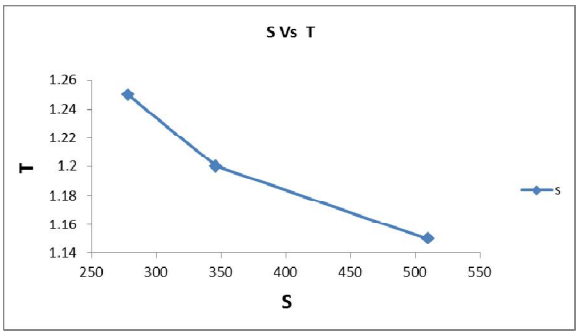

Figure 2: Iinduction period with super saturation for COM

5.Nucleation kinetics of COM crystal

Based on the equations given in the discussion of classical theory of nucleation [7,8, 9], Nucleation parameters like interfacial energy, number of molecules in the critical radius, Gibbs free energy per unit volume (ΔG_v) and critical free energy barrier (ΔG^*), were determined. Interfacial energy is calculated from the slope of ln τ against 1/ $(\ln(s))^2$ plot. The variation of interfacial energy with super saturation is shown in table; it is observed that the interfacial energy decreases gradually with increases in the super saturation of the solution. The measured interfacial energy values vary in the range of 7.519-8.400610^{-3} J/m^2.

The evaluated value of critical radius as a function of super saturation suggests that the free energy changes exponentially with super saturation. The energy barrier, numbers of molecules are found to decrease with super saturation ratio. It is evident from the table as the level of super saturation increases, critical energy barrier and number of molecules decreases. It is worth to note that the minimum nucleation barrier occurs at the interfacial energy of 8.4006 10^{-3} J/m^2. The results are tabulated in table 1.

Super saturation ratio	**Interfacial energy (mJ/cm^2)**	**Critical radius r*(nm)**	**Critical energy barrier ΔG***	**Critical volume V (nm)^3**	**Number of molecules in nucleus i***
1.15	8.4006	104.7131	3.8563	4806	33
1.2	7.9361	82.5868	2.2373	2358	16
1.25	7.519	69.3237	1.5128	1394	10

Table 1: Nucleation Parameters of COM

The present study confirms that the evaluated nucleation parameters are feasible for the growth of bulk COM single crystal [10, 11].

6.Conclusion

Artificial kidney stone i.e. Calcium Oxalate Monohydrate crystals are grown using solvent evaporation method. An attempt has been made to understand the solubility of crystals with different temperatures. The induction periods are evaluated experimentally for crystallization. In addition, for the first time in the literature, the nucleation kinetics of kidney stones are analysed and results are tabulated. On the basis of the obtained nucleation data, single crystals were successfully grown.

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