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ORIGINAL ARTICLE

GROWTH AND THERMAL CHARACTERIZATION OF CHOLESTEROL CRYSTALS

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ABSTRACT

Cholesterol ($C_{27}H_{46}O$) is the most abundant and best known steroid in the animal kingdom. In the present study the crystallization of pure cholesterol monohydrate crystals in gel medium using acetone solvent. In human body, precipitation of cholesterol gallstones occurs due to a defect of crystallization inhibiting or an abundance of crystallization promoting factors. The physical/ chemical events useful method for the identification of factors delaying or preventing precipitation of cholesterol crystals and therefore, gallstone formation in humans .The Gel grown cholesterol crystals are characterized by using TG/DTA,FT-IR and SEM-EDX

Key words: Crystal growth, Cholesterol, gel method, organic solvents, TG/DTA,FT-IR and SEM-EDX

1.INTRODUCTION

Cholesterol is a major constituent of crystalline material in gallstones[Seethalakshmi Ammal et al., 2007; Elizabeth et al., 2001]. Cholesterol has low solubility in water but it is soluble in organic solvents such as ethanol,acetone, methanol and benzene. Cholesterol was crystallized from acetone as a solvent and the effect of solvent on the crystal structure was studied. Cholesterol crystal has fibrous like morphology in silica gel in acetone as a solvent[Kanchana and Sekar,2011; Bruice and Rajendra Prasad,2008]. Gel is an ideal medium to grow biological crystals since its structure is similar to the mucus in the living organisms[Bhagat and Popalghat,2013; Ira et al., 2001; Kalkura and Devanarayanan,1986; Sandarac et al., 2002; Patel and Rao,1982} The internal surface of the organs in animals is invariably covered with mucus membrane. This material has open structure containing pores of different sizes. These pores can act as nucleation centers for the growth of crystals. Even at low super saturation, specific molecules can segregate creating critical nuclei to enhance the growth of crystalline materials. In the present work cholesterol is grown in silica gel medium using acetone as a solvent. The effect of temperature and concentration on the growth is also studied.

2. MATERIALS AND METHODS

The single test-tube diffusion method (Henisch 1988) was employed for growing cholesterol crystals in the gel medium.

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To prepare the silica hydro- gel, aqueous sodium meta silicate (SMS) solutions of 1.03 specific gravity was prepared and acetone were mixed in appropriate amount and was acidified by acetic acid so that the PH of the mixture could be set with in 6.0. This mixture was allowed to set in to the gel form for 4 days.

Fig.1 Cholesterol Crystals grown in gel medium

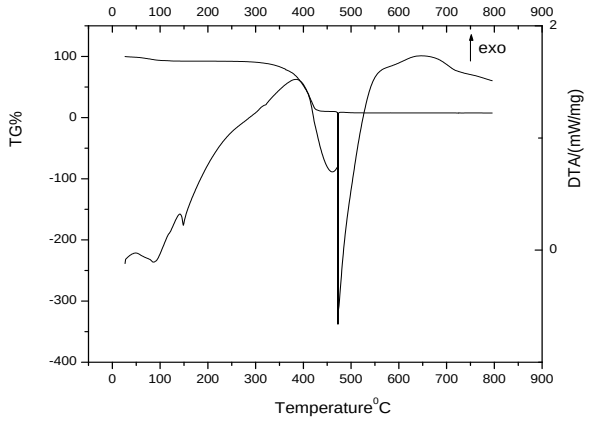
For growing pure cholesterol crystals the supernatant solution was prepared by dissolving 2%(w/v) concentration of cholesterol (purity 99.99%) in acetone solvent and the solution was poured carefully over the top of the silica gel with out disturbing the latter. With in 24 hours fibrous like crystals were found to grow in the supernatant solution . The grown crystals were harvested with in 21 days [Oskokovic ,2008; Sheih et al.,1981; Bhujle et al., 1984].

3. RESULTS AND DISCUSSION

Thermal studies on cholesterol crystals

Fig.2 illustrates TG/DTA curve of pure cholesterol crystals grown from acetone solvent. An endothermic peak seen at a negative peak on DTA trace at 150ºC corresponds to the melting point of cholesterol. Upon further heating, the sample begins to decompose at 360ºC and 90% of mass loss at 418ºC. The mass loss corresponds well with th DTA data , which reveals exothermic peak at 645ºC. There was no further change in the mass until 800ºC.

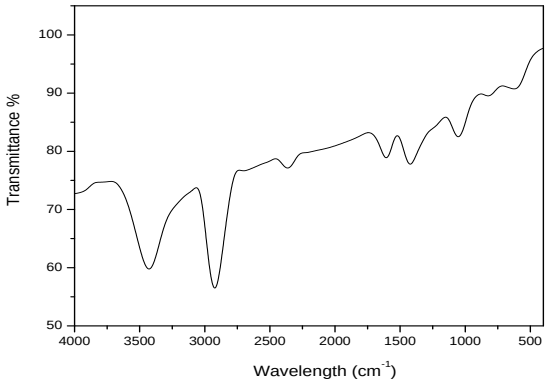
Fig.2 TG/DTA curve of cholesterol crystal



FT-IR spectroscopic studies on Cholesterol Crystals

Fig.3 shows the IR spectrum of cholesterol crystal. FT-IR spectrum cholesterol crystal was recorded SHIMAZDU 400-4000cm^{-1} range in chemistry department at Annamalai university. The respective assignments of the vibrations are given in table1.

Fig.3 FT-IR Spectrum of Cholesterol crystal



The bands observed at 3427 cm^{-1} indicate the presence of water group along with the hydroxyl group which is attached to the cholesterol ring. The bands observed at 2924 region in the spectrum stem from the C-H stretching modes of aromatic compounds. C=O stretching observed at 1606cm^{-1}. C-H deformation band observed at 1421cm^{-1}. The ring deformation of cholesterol can be assigned at 1053cm^{-1}. C-H out -of- plane bending observed at frequency 825cm^{-1}. C-OH-in- plane bend observed at frequency 624cm^{-1}.

Table.1 FT-IR Spectrum of cholesterol crystal

IR Frequency (cm^{-1})	Assignments
3427	O-H strech
2924	C-H stretching
2700	C-H stretching
2364	C-H stretching
1606	C=O stretching
1421	C-H deformation band
1050	Ring deformation
825	C-H out-of-plane bending
624	C-OH in-plane bend

Fig.4 SEM image of cholesterol crystal

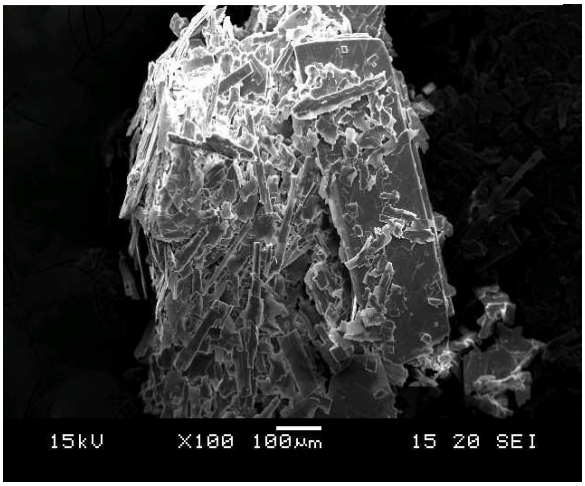
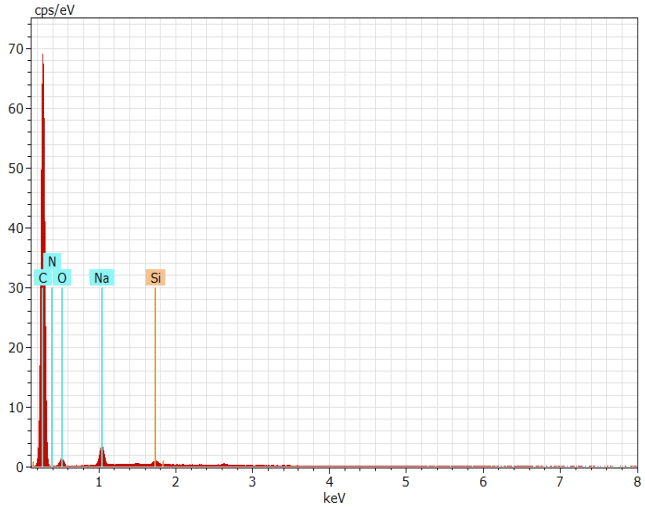


Fig.5 EDX spectrum of cholesterol crystal



SEM with EDX of Cholesterol crystal

Fig. 4 shows SEM analysis of cholesterol crystal. SEM shows plate like morphology of the cholesterol crystal grown in acetone solvent.Fig.5 shows the EDX analysis of the cholesterol crystal.From EDX presence of maximum amount of carbon was identified.

4.CONCLUSION

Fibrous like cholesterol crystals have been grown by gel method using Acetone solvent. TG/DTA confirmed mass loss and thermal stability of cholesterol crystal. FT-IR confirms the functional group present in the cholesterol crystal. SEM with EDX confirmed the morphology and elemental composition present in the cholesterol crystal.

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