The effect of X-Band Microwave Frequencies on the growth of Cholesterol Crystal

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ABSTRACT

The technique of crystals growth has significant role in the recent technology of material science. Various methods have been developed and practiced for the growth of crystals of industrial and commercial interest. The biological organic crystals are also grown by various methods. The study of crystals growth process is also significantly important to understand the growth of various crystals and stones that are grown in human and animal bodies which is major health problem all over the world. In the in vitro crystallization in gel media, the nucleation time, the growth rate and the morphology of crystals depends on various parameters such as solvent used, concentration of solution, specific gravity and temperature. In the present work, the effect of solvent and that of exposure of weak microwave power of X-Band has been studied. The weak microwave is exposed at nucleation state of Cholesterol crystals in gel media and effect is noted for various frequencies and period of exposure. The grown crystals are characterized by using XRD and FTIR method.

Key words: Cholesterol, Silica Gel, Microwave, XRD, FTIR.

INTRODUCTION

The process of crystals growth has been studied by varying various parameters such as solvent, concentration of organic compound in solution, pH of solution and Specific gravity, temperature, presence of organic chemical or bio extract. The change in nucleation time, change in morphology of crystals and change in period of crystals growth are noted by different researchers. The crystal growth in gel media was studied by Patel (Patel *et al.,* 1982). The biological significance of 37˚C phase transition of Cholesterol was studied by Bhujile (Bhujile *et al.,* 1984). The in-vitro Cholesterol crystal has plate like morphology in silica gel in Ethanol solvent as reported by Kalkura (Kalkura *et al.,* 1986). The growth of Cholesterol in different solvents exhibits different morphology and crystal size. (Elizabeth *et al.,* 2001).The observation of Cholesterol nucleation in magnetic field was studied by Sandarac (Sandarac *et al.,* 2002). The change in morphology and nucleation delay after adding extract of phyto active compound in Cholesterol solution in Ethanol solvent was also studied by Ammal (Ammal *et al.,* 2007). We have now studied the effect of X-Band microwave power of various time periods on the crystals growth of Cholesterol, grown in different solvents and compared with growth without exposure of microwave power.

MATERIALS AND METHODS

Crystal growth in gel with exposure of X-band microwave power

The region of nucleation of crystals in gel media is exposed to microwave power. The microwave power is generated by using low power klystron (OTK-5) of 5mW as a source. The various components and their arrangements for igniting microwave power for exposure are shown in block diagram given in figure 1. The exposure of weak microwave power involves four steps, ignition of microwave power using microwave bench, the study of power radiation and power matrix in front of pyramidal horn antenna, gel setting in test tubes and exposing test tube sets in front of pyramidal horn antenna.

Ignition of microwave power using microwave bench

A low power (5mW) klystron tube (OTK-5) is used to expose the Cholesterol crystal growth. The technical specifications are as follows:

Beam Voltage = 275 V
Beam Current = 18 mA
Repeller Voltage = -95 V
Coupler Power = 0.165 mA

The microwave power is obtained in the first mode of klystron by adjusting repellar voltage. The frequency of the exposed microwave power is measured by frequency meter. The frequency of microwave power of klystron can be changed by adjusting tuning screw.

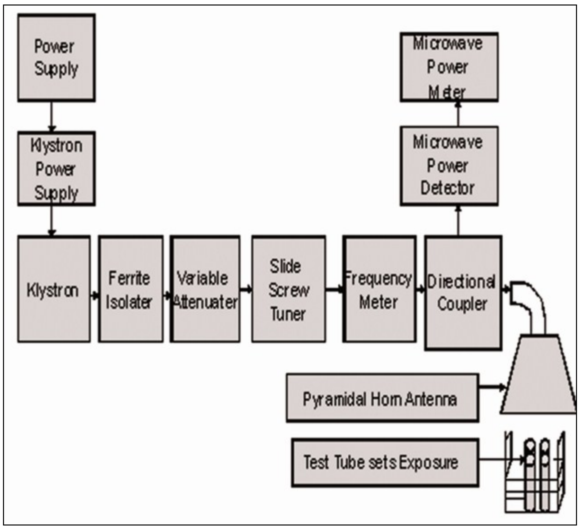

Figure1. Block diagram of Experimental setup for Microwave power exposure.

The study of radiation and power matrix in front of pyramidal horn antenna

To study the microwave power radiation and power distribution at transverse planes at various distances in front of pyramidal horn antenna; the microwave power is ignited in microwave bench setup and fed to antenna. The TE10 mode microwave power can be varied by varying beam voltage and beam current. The maximum power is obtained for the first mode of klystron keeping proper beam voltage and beam current. Care is taken for maintaining the microwave power radiated by horn antenna at constant level during entire period of exposure.

Gel Setting and Cholesterol crystals growth in gel media

The Cholesterol crystals are grown in silica gel media. The gel preparation and gel setting are done by preparing stock solution by dissolving Sodium Meta silicate powder in double distilled water and shaking this solution well. The solution is filtered and kept in clean flask. This solution was mixed with acetic acid to set a suitable pH value of the solution. In the present case pH value is set in (4-6) range and with specific gravity of the solution being 1.035g/cc. Then resulting solution was mixed in organic solvent like Acetone in the ratio 2:1 and allowed to set for 96 hours at room temperature 32˚C. After setting the gel in Acetone solvent, the supernatant solution of 2% concentration of solution in Acetone is poured over set gel. As soon as this solution is added the nucleation process starts at gel formed region of the test tube. Thereafter the test tube should be kept in a quiet and vibration free condition. Fine Cholesterol crystals were observed in test tube after 72 hrs. Similar procedure is repeated for Ethanol and Methanol solvents.

Exposure of microwave at nucleation state of Cholesterol

The microwave power of frequency 9.685 GHz is fed to antenna. The set of four test tubes were prepared for crystal growth in gel media using Acetone solvent as discussed above. The set is kept at 11cms in front of horn antenna so that strength of microwave power level at gel in test tube is around 0.288 mA. Keeping the power level and frequency constant, the first test tube from set is removed after exposure of 30 min., the second test tube from set is removed after exposure of 60 min., the third test tube from set is removed after exposure of 90 min., and the last the fourth test tube is exposed for 120 min. Then all the test tubes are kept for further growth in a quiet condition. Similar procedure is repeated for Ethanol and Methanol solvents for frequencies 9.750 GHz, 9.930 GHz, 9.950 GHz. After 72 hours grown crystals are collected from each test tube on filter paper and studied further. The photographs of Cholesterol crystals grown in Acetone, Ethanol and Methanol solvents without exposure [a, b, c] and with microwave exposed growth [d, e, f] are shown in figure 2.

Crystal Characterization Analysis

XRD and FTIR Studies were conducted to characterize the crystals grown in silica gel media with and without weak microwave power exposure and grown in Acetone, Ethanol, and Methanol solvents.

Powder X Ray Diffraction Analysis

Powder XRD patterns of Cholesterol crystals without microwave exposure and grown in acetone solvent are recorded by Philips PW3710

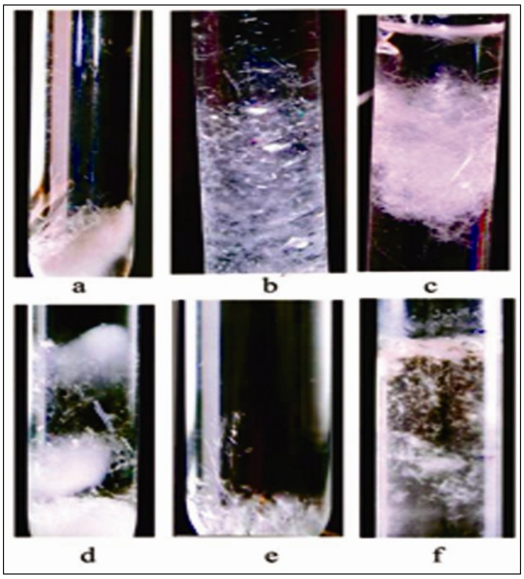

Figure2. Photographs of Cholesterol crystals grown in Acetone, Ethanol and Methanol solvents a), b),c) for without exposed and d), e), f) for microwave exposed growth.

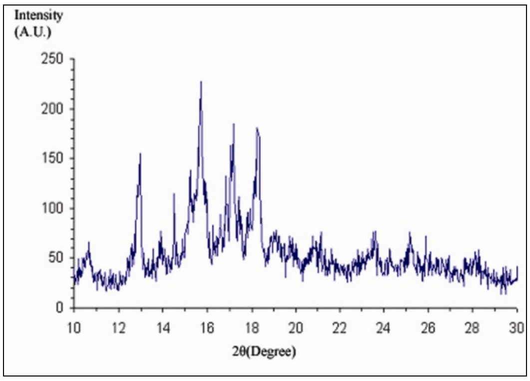

Figure3. XRD of Cholesterol crystals grown without exposure in Acetone solvent.

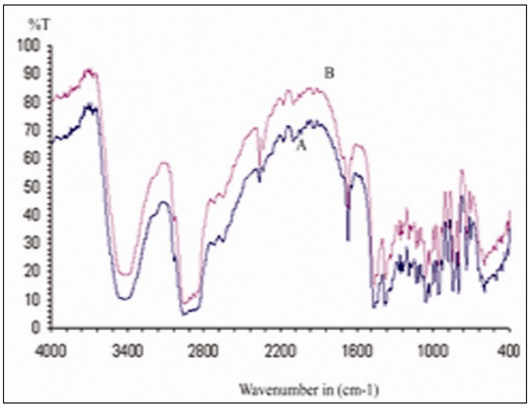

Figure4. FTIR analysis of Cholesterol crystals grown in Acetone solvent without exposure(A) and with microwave exposure(B).

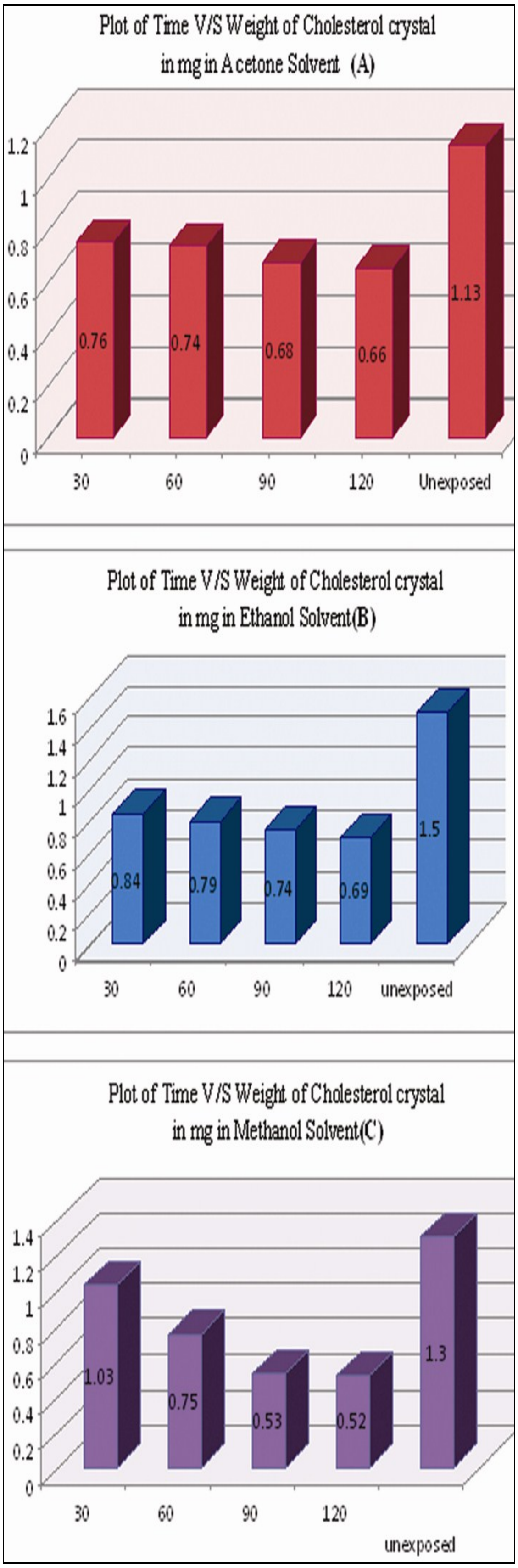

Figure5. Cholesterol Crystals Mass Analysis in A) Acetone B) Ethanol, C) Methanol solvents.

diffractometer using Cu Kα radiation and are shown in figure 3. Cholesterol crystallizes in triclinic structure with cell parameters as follows;

a=14.10A˚, b=33.74A˚, c=10.46A˚,

α=94.60˚, β=90.0˚ γ=95.72˚

FTIR Analysis

The FTIR spectrum of Cholesterol crystals is recorded using FTIR-BUSY-6100JASCO spectrometer in a scan range (4000-400cm^{-1}). The FTIR analysis of crystal grown with and without microwave exposure in acetone solvent is as shown in figure 4. The absorption band observed in (4000-1400cm^{-1}) range is called functional group region and the absorption band observed in range (1400-400cm^{-1}) is called finger print region. It is found that for O-H functional group FTIR spectra recorded for exposed Cholesterol crystals shows shifting of relative frequencies to lower sides in comparison with the FTIR spectra obtained for unexposed crystals in Acetone solvent. For Ethanol and Methanol solvents it shows opposite trend for the same functional group. For C-H functional group FTIR spectra recorded for exposed Cholesterol crystals shows shifting of relative frequencies to higher sides in comparison with the FTIR spectra obtained for unexposed crystals in Acetone solvent. Again for Ethanol and Methanol solvents it shows opposite trend for the same functional group.

Crystals Mass Analysis

Mass of Cholesterol crystals grown for different time of exposure of microwave of power 0.288 mA at frequency 9.930 GHz at nucleation state in Acetone, Ethanol, Methanol solvents are plotted and compared with that of crystals without microwave exposure in figure 5.

RESULTS AND DISCUSSION

The photographs of Cholesterol crystals grown in Acetone, Ethanol and Methanol solvents for without exposed and microwave exposed conditions are shown in figure 2.

The XRD of Cholesterol crystals grown without exposure in Acetone solvent are shown in figure 3. Our results are in good agreement with results reported in literatures (Sheih,etal, 1981, Kanchana, etal, 2011).

The FTIR analysis of crystals grown without exposure (A) and with weak microwave exposure (B) in Acetone solvent is shown in figure 4. The results are in good agreement with results published (Uskokovic,2008, Kanchana, etal,2011, Begum *etal.*,2012).

The morphology of Cholesterol Crystals observed as rectangular, plate like in Acetone solvent, thin Plate like and lath like in Ethanol solvent and needle like in Methanol solvent are in good agreement with result published.

Mass of Cholesterol Crystals grown without expose and exposed to weak microwave power in Acetone, Ethanol and Methanol solvents and their comparison are shown in figure 5.

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