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# A Comparison of 95 MeV Oxygen Ions and Co-60 Gamma Irradiation Effect on Nonlinear Optical L-Alanine Cadmium Chloride (LACC) Single Crystals

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**Abstract:** Single crystal of nonlinear optical (NLO) L-alanine cadmium chloride (LACC) was grown by slow evaporation method at room temperature. The grown crystals were irradiated by 95 MeV oxygen ions and Co-60 gamma radiation at doses of 1 Mrad and 6 Mrad. The structural, chemical, dielectric properties, AC and DC conductivity, refractive index (RI) and second harmonic generation (SHG) of the crystals were studied before and after irradiation. The slight change in the lattice parameters was observed due to compressive strain field generated in the irradiated crystals. There is no significant formation of intermediate chemically distinct material after ion and gamma irradiation. The observed increase in dielectric constant was found to be more for Co-60 gamma irradiated crystals than 95 MeV oxygen ions irradiated crystals. The AC and DC conductivity were found to increase after irradiation. A considerable change in the values of RI was observed for both ion and gamma irradiated crystals. The SHG efficiency of LACC crystals was found to decrease with increase in radiation dose.
**Keywords:** Ion irradiation, gamma irradiation**,** nonlinear optical, irradiation effect, SHG.

## 1. Introduction

After the advent of laser, the nonlinear phenomena made a big revolution in the field of optics and the frequency conversion become an important and popular for laboratory lasers (1-3). Many of solid-state lasers are designed to work in the strong external fields of ionizing radiation. Exposure of laser materials to space radiation and to high-energy photons (>30 MeV) can damage due to ionization of atoms and structural damage of the laser material (4-6). Therefore understanding the effects of different radiations on solid state materials in particular NLO crystals is an important problem and has practical applications. When high energy swift heavy ion passes through matter; it loses its energy mainly in two ways. The interaction of heavily charged ions with electrons of the target material produces a track of ionization and highly kinetic electrons along the path of the primary ion due to inelastic collision. This is known as electronic energy loss or electronic stopping. Nuclear energy loss or nuclear stopping which is caused by the elastic scattering from the nuclei of the atoms. It is dominant near the end of the range of implanted ions and spent in displacing atoms of the sample (7-14). It is known that gamma rays produce radiation damage via the creation of Compton electrons which can cause further ionizations and produce secondary electrons which produce point defects in bulk of the material in addition to ionization and in turn altering the material properties (5, 15). We present, for the first time, a systematic comparison of 95 MeV $O^{7+}$ ions and Co-60 gamma irradiation on structural, chemical, dielectric properties, AC and DC electrical conductivity, refractive index and SHG efficiency of nonlinear optical LACC crystal.

## 2. Experimental Details

The single crystal of LACC was synthesized from L-alanine and cadmium chloride monohydrate taken in the equi-molar ratio. The calculated amounts of the reactants were dissolved in double distilled water and stirred well for about 2 hours using a magnetic stirrer at 30$^oC$ to form a saturated solution. The solution was then filtered twice to remove the suspended impurities and allowed to crystallize by slow evaporation of solvent at room temperature. Good transparent crystals of size around 3.4 cm x 3 cm were obtained in a period of about four weeks and are shown in figure.1. The crystals were exposed to 95MeV $O^{+7}$ ions at the 15 UD 16 MV Pelletron Tandem Van de Graff Accelerator at Inter University Accelerator Center (IUAC), New Delhi, India. The experiments were performed at 300 K in the experimental chamber of diameter 1.5 m maintained at $10^{-7}$ mbar vacuum, with the ion fluence $2.1\times10^{10}$ ions/cm$^2$ and $1.3\times10^{11}$ ions/cm$^2$ and its equivalent gamma doses are 1 Mrad and 6 Mrad respectively. The typical beam current during irradiation was 0.286 particle nano-ampere (pnA). The incident ions have a range of penetration of 150 µm in the crystal as determined by SRIM-2008 calculations. The electronic energy loss in LACC crystals is 2947 MeVcm$^2$/g, whereas nuclear energy loss is 1.689 MeVcm$^2$/g. LACC crystals were exposed to Co-60 gamma radiation using gamma chamber 5000 at Central Instrumentation Facility, Pondicherry University, India with the dose rate of 167 rad/sec and average gamma energy of 1.25 MeV at doses of 1 Mrad and 6 Mrad. The total dose was kept identical for both high energy ions and gamma radiation.

**Figure1. The photograph of the as-grown LACC single crystals**

## 3. Results and Discussion

### 3.1. X-Ray Diffraction Analysis

The single crystal X-ray diffraction of as grown, 95 MeV $O^{7+}$ ions and Co-60 gamma irradiated LACC crystals was done using Nonius CAD-4/MACH3 diffractometer, with Mo-Kα radiation (λ=0.71073 Å). From the single crystal X-ray diffraction data (Table 1), it can be seen that there is no change in the phase structure of the irradiated samples; however there is a slight change in the lattice parameters and this may be due to the compressive strain field generated in the irradiated crystals (16,11,17).

**Table 1 The lattice parameters of the unirradiated and irradiated LACC crystals**

| Lattice parameters | Unirradiated | 95 MeV oxygen ion | | Co-60 gamma | |
|---|---|---|---|---|---|
| | | 1 Mrad | 6 Mrad | 1 Mrad | 6 Mrad |
| a (Å ) | 16.415 | 16.400 | 16.111 | 16.305 | 07.982 |
| b (Å) | 7.279 | 7.254 | 7.283 | 7.249 | 7.283 |
| c (Å) | 07.989 | 07.989 | 07.924 | 07.993 | 14.601 |
| β (◦) | 116.53 | 116.40 | 116.38 | 116.67 | 092.77 |
| volume (Å$^3$) | 854.0 | 853.1 | 851.6 | 844.2 | 848.2 |
| Crystal structure | Monoclinic | Monoclinic | Monoclinic | Monoclinic | Monoclinic |

### 3.2. FTIR Spectral Analysis

The infrared spectral analysis provides useful information regarding the molecular structure and functional groups of the compound. The infrared spectrums of unirradiated and irradiated LACC crystals [figure.2] were recorded in the frequency range 400–4000 cm$^{-1}$ using FTIR-8400S spectrophotometer, SHIMADZU model under a resolution of 4 $cm^{-1}$ and with the scanning speed of 2 mm/sec. The $NH_2$ group of L-alanine is protonated by the COOH group. The presence of $NH_3^+$ is very easily identified in the FT-IR spectrum by the broad intense band with the strong absorption at 3229 $cm^{-1}$ and 3081 $cm^{-1}$ corresponding to asymmetric and symmetric stretching mode of $NH_3^+$. The $NH_3^+$ symmetric stretching frequencies are overlapping with the vibrations of $CH_3$ group. The strong absorption at 1464 $cm^{-1}$ indicates the bending degenerate frequency of the carbonyl group. The absorption at 1187 $cm^{-1}$ and 844 $cm^{-1}$ corresponding to asymmetric and symmetric stretching mode of CCN. The $COO^-$ rocking and bending frequencies occur at the 1431, 763, 633 and 534 $cm^{-1}$. The absorption peaks at 920 $cm^{-1}$ and 843 $cm^{-1}$ are assigned to C–C–N symmetric stretching vibrations. These vibrations prove the presence of expected functional groups in the compound (18, 19). It can be seen from figure.2 that, there are some of the absorption bands completely destructed after irradiation which enhance the amorphisation in the sample (20). The absence of a prominent new peak in irradiated crystals confirms that there is no significant formation of intermediate chemically distinct material during irradiation (16, 21).

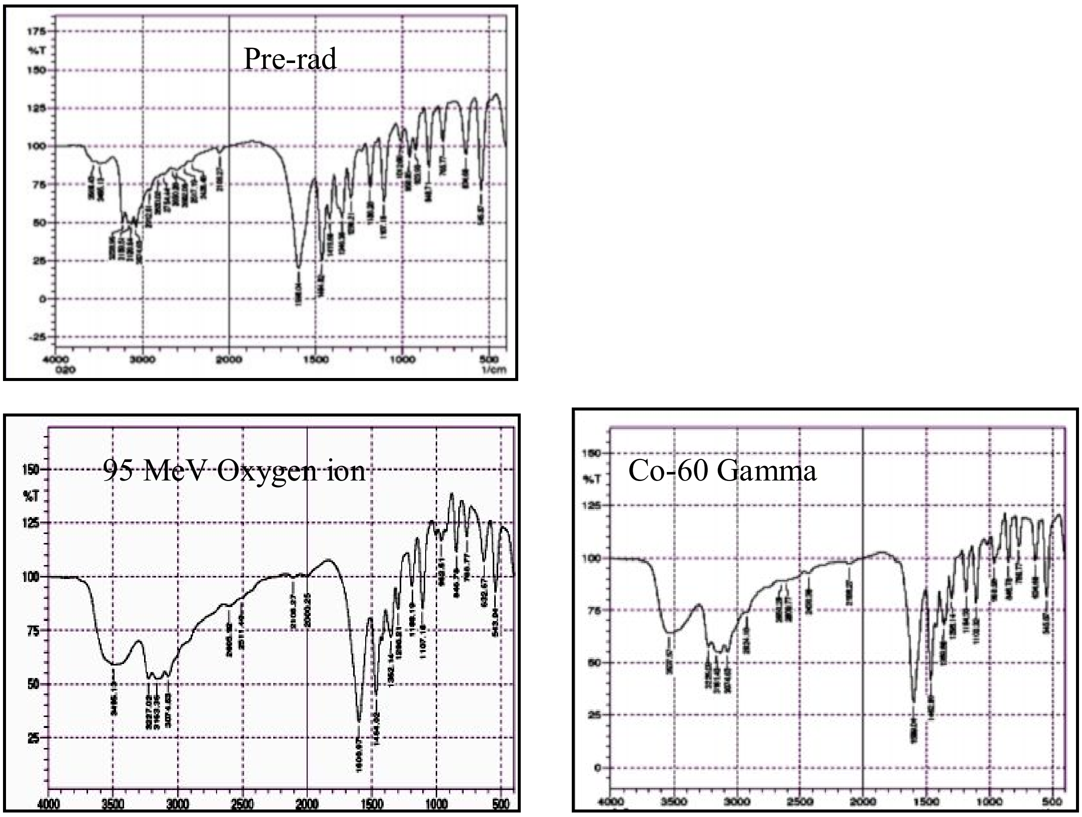


**Figure. 2. FT-IR spectra of unirradiated and irradiated LACC crystals (after 6 Mrad)**

### 3.3. Dielectric measurement

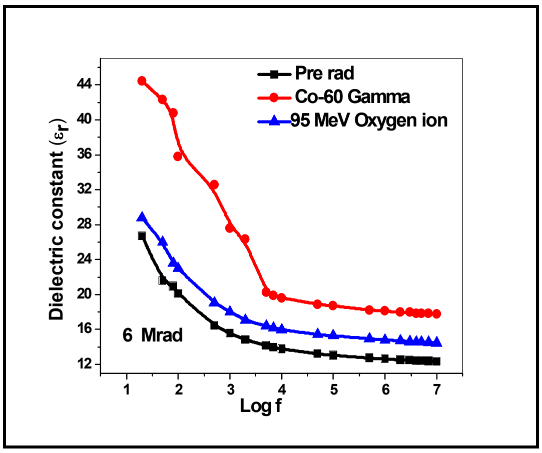


**Figure 3. Plot of dielectric constant versus applied frequency for unirradiated and irradiated LACC crystals (after 6 Mrad)**

Dielectric measurement is one of the useful methods for characterization of electrical response in crystalline materials. The surfaces of the samples were polished and coated with silver past to ensure good electrical contacts. The capacitance and dissipation factor of the unirradiated and irradiated crystals have been measured using High Frequency LCR Meter 6500P in the frequency range from 20 Hz to 10 MHz. The dielectric constant $\varepsilon_r$ has been calculated using the equation:

$$\varepsilon_r = \frac{Cd}{A\,\varepsilon_0} \qquad (1)$$

where $\varepsilon_0 = 8.854\times10^{-12}$ F/m is the permittivity of free space, $d$ is the thickness of the sample, $C$ is the capacitance and $A$ is the area of the sample. Figure.3 shows the plot of the dielectric constant versus applied frequency for unirradiated, 95 MeV $O^{7+}$ ions and Co-60 gamma irradiated LACC crystals. From the figure, it can be seen that the dielectric constant was found to decrease with increase in frequency before and after irradiation. The large values of dielectric constant at low frequency might be attributed to the presence of space charge polarization. The decrease in the values of dielectric constant with the frequency takes place when the jumping frequency of electric charge carriers cannot follow the alternation of the ac electric field applied beyond a certain critical frequency (9, 11, 12, 14). The dielectric constant was found to increase after irradiation. The increase in dielectric constant may be correlated to the defects created after irradiation and also may be attributed to the resultant disordering of the irradiated crystal lattice. These defects cause an increased space-charge contribution which increase the dielectric constant of crystals (4-12, 14, 15). The observed increase in dielectric constant was found to be more for Co-60 gamma irradiated crystals than 95 MeV $O^{7+}$ ion irradiated crystals, since gamma radiation can generate more defects when compared to the high energy ions. The dielectric loss may be due to the perturbation of the phonon system by an electric field. The energy transferred to the phonons dissipates in the form of heat (9, 12, 13, 22). The variation of dielectric loss as a function of frequency is shown in figure.4. From the figure, it can be seen that the dielectric loss was found to decrease after irradiation.

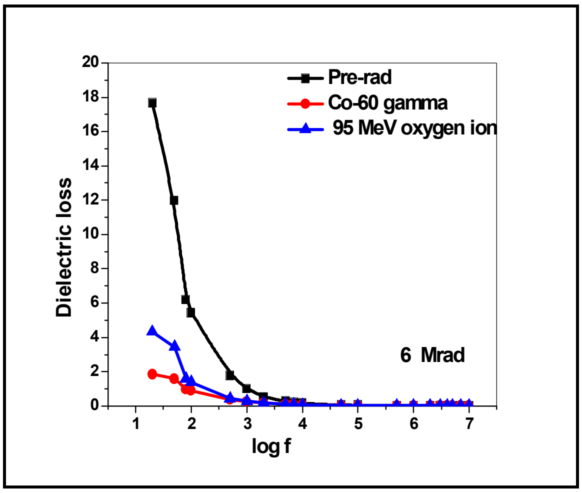


**Figure 4. The Variation of dielectric loss as a function of frequency for unirradiated and irradiated LACC crystals (after 6 Mrad)**

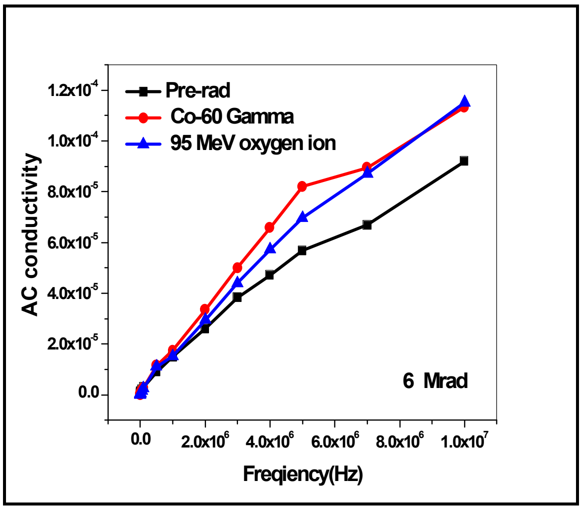


**Figure 5. Variation of ac conductivity versus frequency for unirradiated and irradiated LACC crystals (after 6 Mrad)**

The ac conductivity $\sigma_{ac}$ is calculated by substituting the value of dielectric constant $\varepsilon_r$ and dielectric loss $\tan\delta$ in the relation:

$$\sigma_{ac} = 2\pi f \varepsilon_0 \, \varepsilon_r \tan\delta \qquad (2)$$

where $f$ is the frequency of the applied field. Figure.5 shows the response of ac conductivity with frequency in the range from 20 Hz to 5 MHz for unirradiated, 95 MeV $O^{7+}$ ions and Co-60 gamma irradiated LACC crystals. From the figure, it can be seen that ac conductivity was found to increase with increase in frequency before and after irradiation. The observed ac conductivity was found to be more at higher frequencies due to a reduction in the space charge polarization. The ac conductivity of the crystal was found to increase after irradiation owing to the fact that more defects are created upon irradiation (16, 14, 23-26).

### 3.4. Dc Conductivity Study

**Table 2 DC conductivity values of unirradiated and irradiated LACC crystals**

| | Unirradiated | 95 MeV oxygen ion | | Co-60 gamma | |
|---|---|---|---|---|---|
| | | 1 Mrad | 6 Mrad | 1 Mrad | 6 Mrad |
| Conductivity $(\Omega.\ m)^{-1}$ | $4.92\times 10^{-10}$ | $6.36\times 10^{-10}$ | $5.24\times 10^{-9}$ | $1.29\times 10^{-9}$ | $3.71\times 10^{-8}$ |

The dc conductivity $\sigma_{dc}$ of the unirradiated and irradiated LACC crystals was calculated using the relation:

$$\sigma_{dc} = \frac{d}{RA} \qquad (3)$$

where $R$ is the measured resistance (25, 27-29). The dc conductivity measurement was carried out using Keithley dual channel source meter model 2636A at different temperatures ranging from 300 to 383 K. The I-V characteristic curves of unirradiated, 95 MeV $O^{+7}$ ions and Co-60 gamma irradiated LACC crystals are shown in figure.6 and from which the conductivity values were calculated. The dc conductivity values are given in Table 2. From the table 2, it can be seen that, the dc conductivity was found to increase with increase in irradiation dose owing to the fact that more defects are created upon irradiation and hence the conductivity (which is predominantly due to the increase in the movement of defects) increases (16, 14, 23-26). The observed increase in the dc conductivity was found to be more for Co-60 gamma irradiated crystals than 95 MeV $O^{7+}$ ion irradiated crystals. Figure.7 shows the plot of dc conductivity with temperature for unirradiated, 95 MeV $O^{7+}$ ions and Co-60 gamma irradiated LACC crystals. From this figure, it can be seen that the dc conductivity was found to increase with increase in temperature before and after irradiation.

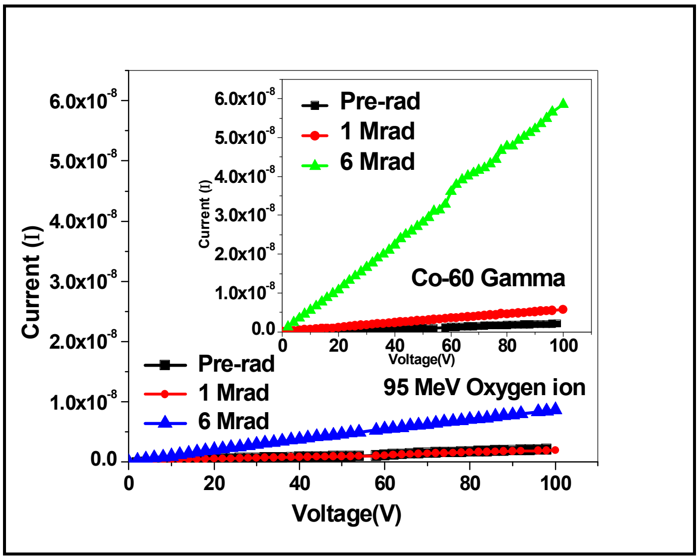


**Figure.6. I-V graph of unirradiated and irradiated LACC crystals**

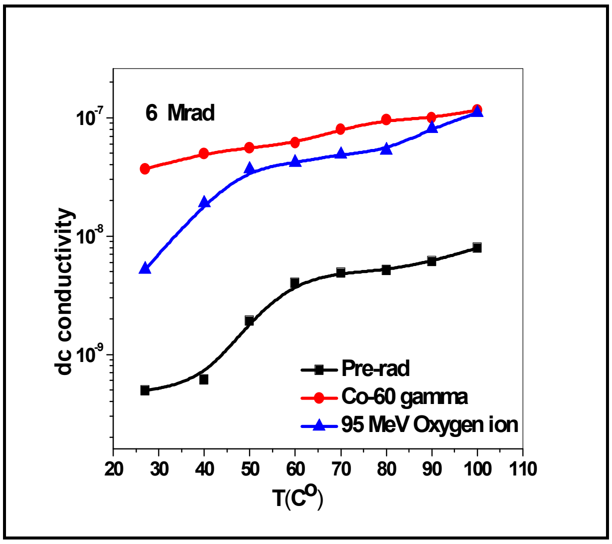


**Figure 7. Variation of dc conductivity versus temperature for unirradiated and irradiated LACC crystal (after 6 Mrad)**

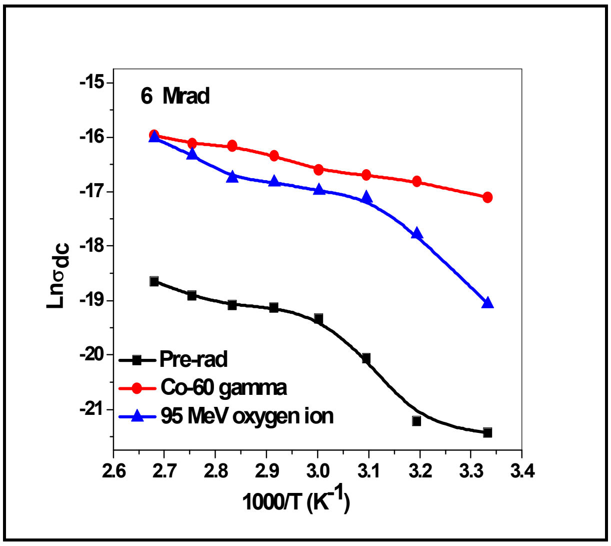


**Figure 8. Plot of $Ln\sigma_{dc}$ versus $\frac{1000}{T}(K^{-1})$ for unirradiated and irradiated LACC crystal (after 6 Mrad)**

The conductivity graph exhibits the intrinsic and extrinsic regions. Conductivity at high temperature above 310 K is intrinsic, which is due to the thermally created vacancies and defects created in crystalline lattice. Extrinsic region at low temperatures les than 310 K is a structure-sensitive region i.e., electrical conductivity is controlled by impurities (23, 25, 26, 28-30). The activation energies for unirradiated and irradiated samples were calculated from figure.8 using the equation:

$$\sigma = \sigma_0 \exp(\frac{E_a}{K_B T}) \quad (4)$$

where $E_a$ is the activation energy, $K_B$ the Boltzmann constant, $T$ the absolute temperature and $\sigma_0$ is the constant depending on the material. The activation energies obtained for unirradiated and irradiated LACC crystals are tabulated in Table 3.

**Table 3** Activation energy of the unirradiated and irradiated LACC crystals (after 6 Mrad)

| Crystal | Activation energy $E_a$ (eV) |
|---|---|
| Unirradiated | 0.3915 |
| 95 MeV oxygen ion | 0.3457 |
| Co-60 gamma | 0.1506 |

### 3.5. Refractive Index (RI) Measurement

Brewster's angle method was used to study the RI of unirradiated and irradiated LACC crystals by using a red (He-Ne) laser of 633 nm wavelength. The refractive index 'n' of unirradiated, 95 MeV $O^{7+}$ ions and Co-60 gamma irradiated LACC crystals were calculated using the equation:

$$n = \tan\theta_p \quad (5)$$

where $\theta_p$ is the polarizing angle and the results are tabulated in Table 4.

**Table 4 Refractive index of the unirradiated and irradiated LACC crystals**

| Crystal | Polarizing angle(degree) | Refractive index ("n)" |
|---|---|---|
| Unirradiated | 55.65 | 1.463 |
| 95 MeV oxygen ion | 56.30 | 1.499 |
| Co-60 gamma | 56.80 | 1.528 |

From the table 4 it can be seen that, the RI was found to increase after irradiation. The increase in RI may be correlated to the defects created after irradiation. These defects offer high refraction for the light traveling through irradiated crystal (7, 8, 31, 21). The observed increase in RI was found to be more for Co-60 gamma irradiated crystals than 95 MeV oxygen ion irradiated crystals. The modification in the RI due to irradiation implies the possibility of fabricating optical waveguides in these crystals (10, 31-33).

### 3.6. SHG Conversion Efficiency Measurement

The study of nonlinear optical conversion efficiency has been carried out using the modified setup of Kurtz and Perry (34) at the Indian Institute of Science, Bangalore. To study the radiation induced effect on nonlinear properties of LACC crystals, the SHG efficiency of irradiated crystal was compared with that of unirradiated crystal. A Q-switched Nd:YAG laser beam of wavelength 1064 nm was used with an input power of 2.15 mJ and pulses width of 10 ns with a repetition rate of 10 Hz. The crystals were powdered and then packed in a micro-capillary of uniform bore and exposed to laser radiations. Second harmonic radiation generated by the randomly oriented microcrystals was focused by a lens and detected by a photomultiplier tube. The optical signal incident on the PMT was converted into voltage output at the cathode ray oscilloscope. The comparison of SHG conversion efficiencies of unirradiated, 95 MeV $O^{7+}$ ion and Co-60 gamma irradiated crystals are given in Table 5. The results show that SHG conversion efficiency of LACC crystals were found to decrease with increase in radiation dose. The observed decrease in SHG efficiency was found to be more for Co-60 gamma irradiated crystals than 95 MeV $O^{7+}$ ion irradiated crystals. This indicates that irradiation decreases the laser damage threshold of LACC crystal (6, 35, 31).

**Table 5 Comparison of SHG conversion efficiency for unirradiated and irradiated LACC crystals**

| | Unirradiated | 95 MeV oxygen ion | | Co-60 gamma | |
|---|---|---|---|---|---|
| | | 1 Mrad | 6 Mrad | 1 Mrad | 6 Mrad |
| Second harmonic signal (mV) | 9.5 | 9.0 | 7.0 | 5.5 | 4.4 |

## 4. Conclusions

The structural, chemical, dielectric properties, AC and DC conductivity, refractive index and SHG efficiency of LACC crystals were studied before and after exposure to 95 MeV $O^{7+}$ ions and Co-60 gamma radiation at doses of 1 Mrad and 6 Mrad. We found that there is no structural phase change in the crystals due to irradiation, however there is a slight change in the lattice parameters due to compressive strain field generated in the irradiated crystals. The observed increase in dielectric constant was found to be more for Co-60 gamma irradiated crystals than 95 MeV $O^{7+}$ ion irradiated crystals. AC conductivity strongly depends on frequency and at high frequencies, the conductivity increases, and is supported by a decrease in the dielectric constant at high frequencies. The present study indicates that the dc conductivity increases with the increase in radiation dose and temperature. The observed increase in the dc conductivity was found to be more for Co-60 gamma irradiated crystals. Refractive index of LACC crystal was found to increase after ion and gamma irradiation. The decrease in SHG efficiency after irradiation was found to be more for Co-60 gamma irradiated crystals than 95 MeV $O^{7+}$ ion irradiated crystals.

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