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Growth and optical properties of organic GOA crystals

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Abstract: - A new organic NLO crystal GOA was synthesized by slow evaporation method .Glycine and oxalic acid were combined in 3 different molar ratios to form glycine oxalic acid (GOA)transparent crystals of sizes (2-6 cm) in 2 to 3 weeks time. The phase formation using XRD studies shows orthorhombic crystal structure. The UV visible studies shows wide transparency window between 188 nm to 700 nm suggesting the use of grown materials for non linear applications. The NLO studies using Nd:YAG laser shows appreciable conversion efficiency for sample with change in concentration of oxalic acid. The samples exhibited positive as well as negative photoconductivity at lower applied fields, with more resolution at higher applied fields>200 volts/cm, separation between the light and dark current have been observed.

Keywords: - *Glycine, Oxalic acid ,UV studies, NLO studies, Photoconductivity studies*

I. INTRODUCTION

Crystal growth is a vital and fundamental part of material sciences and engineering. In the field of molecular electronics the organic material are demanded because of their versatile functionalities. The intermolecular interactions in organic materials affect novel functionalities such as conducting, dielectric and optical properties. In organic materials charge transfer process i.e transfer of electron between donor and acceptor molecules play critical roles. New organic NLO crystals are being developed with the physic-chemical stability of organic crystals with good second harmonic generation efficiency by increasing the intermolecular interactions. The main importance of organic NLO materials is that they are used in optical devices because of their large optical nonlinearity, low cutoff wavelength, short response time and high threshold for laser power. The organic NLO crystals are synthesizes with non-localized π electron system to realize nonlinear susceptibility better than inorganic crystals [1-4].Organic NLO materials are the key elements for future photonic technologies.The main advantage of organic materials is that they can be modified and tuned with respect to their chemical structure and properties of materials according to our requirement i.e. they have large structural diversity[5-7].The nonlinear optical (NLO)properties of organic materials are currently of great interest for applications in the field of communication technologies. Owing to the tendency to replace classical electronic material with suitable organic compound, organic crystals have non-linearity optical coefficient larger than inorganic crystals. In the field of nonlinear optical crystal growth, amino acids playing a vital role. A number of natural amino acids exhibit the nonlinear optical properties because they have a donor NH_2 and acceptor COOH and also intermolecular charge transfer is possible[8-9]. Among the amino acids, glycine is the basic one. It has no asymmetric carbon and is optically inactive. The Hydropathy index of glycine is -0.4. The second harmonic generation (SHG) variation of glycine based compounds is due to the interaction of acceptor and donor groups. The interaction between the NH_2 group (which is donor) and the –COOH (which is acceptor) results in the variation of SHG efficiency. Glycine with oxalic acid forms the crystals of glycine oxalic acid (GOA).The different molar proportions of glycine and oxalic acid results in the formation of a series of GOA crystals. It is interesting to grow new GOA (Glycine oxalic acid) series of crystals having NLO Characteristics[10] Glycine oxalic acid (GOA) crystals have been successfully grown by slow evaporation of solution containing the stoichiometric ratios of its components. The amino acid glycine (NH_2CH_2COOH) is the only protein forming amino acid without centre of chirality's [11]. Grown crystals were characterized by various techniques. The present paper gives insight of comparative analysis of the crystals with respect to its structure and optical properties.

II. EXPERIMENT

2.1 Growth of GOA crystals

Analytical reagent grade (AR) samples of glycine (NH_2CH_2COOH) and Oxalic acid $(COOH)_2$ (anhydrous) were dissolved in double distilled water and used for growth of crystal by slow evaporation method at room temperature. Glycine and oxalic acid were taken in 3 different molar ratios, viz 3:1,2:1,1:1 and saturated so;tion were filtered using WHATMAN 110 µm filter paper. All the crystals were found to be stable, colorless and transparent (Fig1).The period of growth ranged from about 2 to 3 weeks.

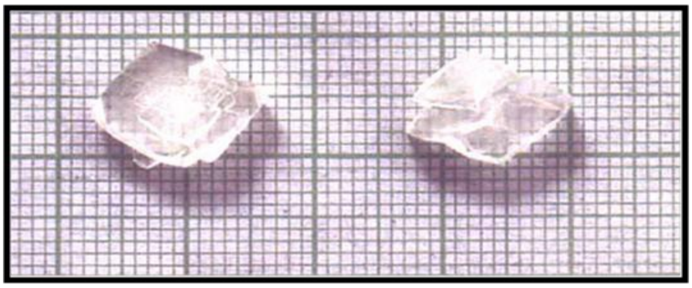

Figure 1 .Photograph of GOA series

III. RESULT AND DISCUSSION

3.1 Powder X-ray Diffraction Analysis

X-ray diffraction (XRD) is a well established technique for determine crystalline order in 3 dimensional solid. The crystals in powder X-ray diffraction technique exposed to characteristics X-ray (CuKα wavelength λ=1.5418A0) and output is obtained in the form of x-ray peaks where the Bragg's law is satisfied(fig2). The GOA crystals belong to orthorhombic symmetry. The cell parameters are dependent on the concentration of oxalic acid in the crystals (Table1).A software program was used to assign planes and determine the lattice parameters for all the grown crystals.

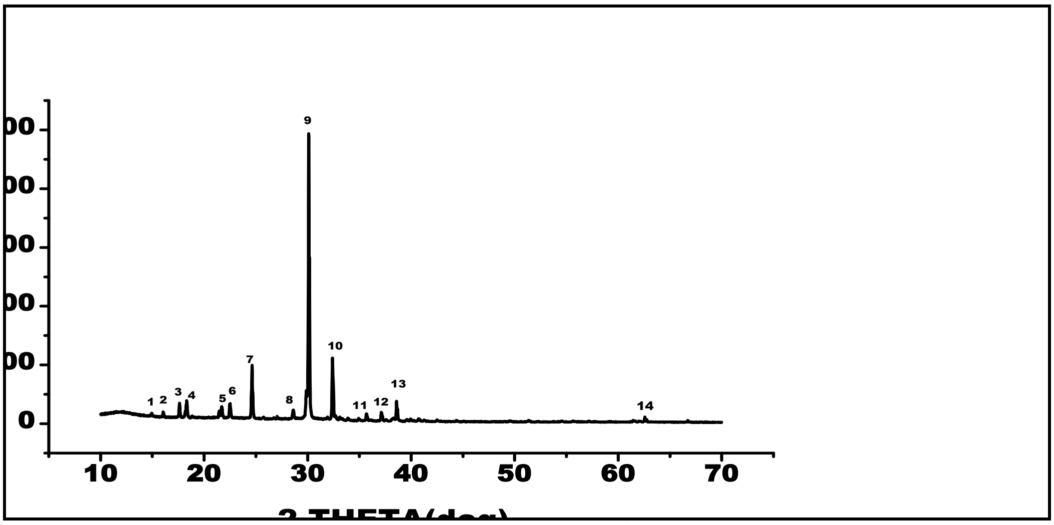

Figure 2 xrd pattern for GOA 1

Table 1 : X-Ray Diffraction - Lattice parameter values

Sample	a (A^0)	b (A^0)	c (A^0)	Volume	Structure
GOA 1	7.035	12.20	9.05	777.33	Orthorhombic
GOA2	9.02	10.01	14.50	1309.21	Orthorhombic
GOA3	17.50	23.10	26.43	10707.45	Orthorhombic

3.2 Ultra Violet Spectral studies

In UV spectral studies the optical transmittance window, the transparency and the lower cutoff wavelength is very important for the realization of SHG output in the range using diode laser. When the samples are transparent, an attempt is made to record the region in which they behave as nonlinear optical material. The ultra violet spectra were therefore recorded from 150nm to 700nm.If the maximum wavelength i.e. λ_{max} lies within this region, the existence of wide transparency window indicates material of NLO properties and which find applications in electronic industries[12-13].The spectral profiles showed that the samples are transparent within the region 190 nm to 700 nm (Fig 3 to5).The maximum cut-off wavelength of GOA (series) varies as the concentration of oxalic acid changes with respect to glycine, the λ_{max} value remains unchanged whereas the absorbance power is seen to decrease with increase in concentration of oxalic acid (figure 6). The effect of concentration of oxalic acid on λ_{max} and absorbance power are summarized in Table 2.

Table2-λmax and absorbance of GOA

Sample	Concentration of Oxalic acid (%)	Cutoff Wavelength (λ_{max}) nm	Absorbance
GOA 1	0.33	188.0	1.761
GOA 2	0.50	188.0	1.523
GOA 3	1.00	188.0	0.572

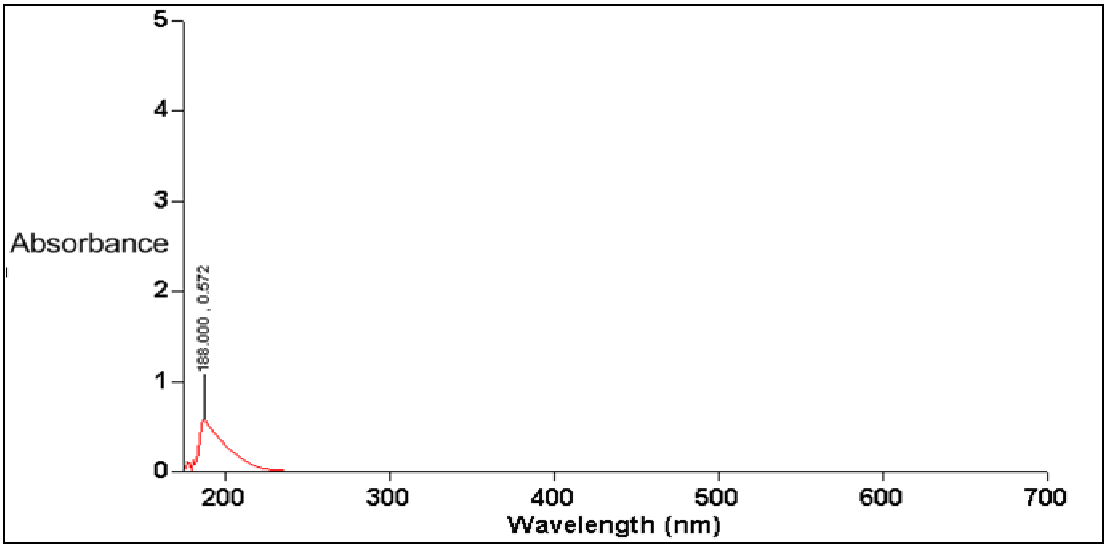

Figure3. UV spectrum for GOA 1

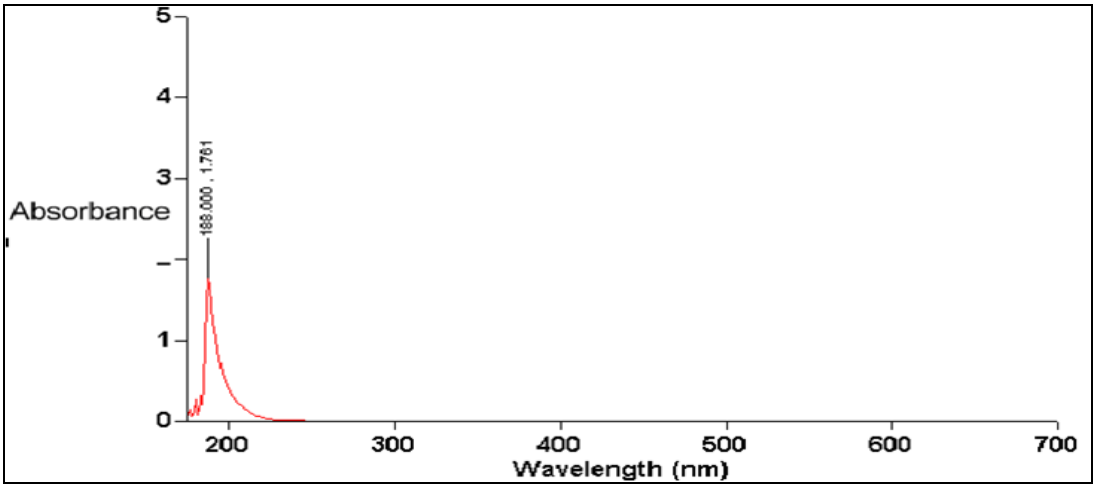

Figure 4. UV spectrum for GOA 2

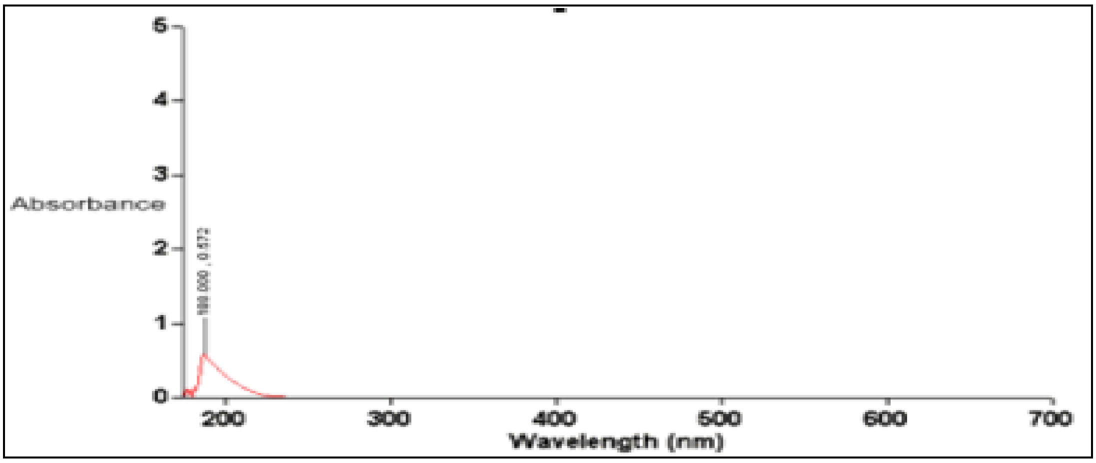

Figure5. UV spectrum for GOA 3

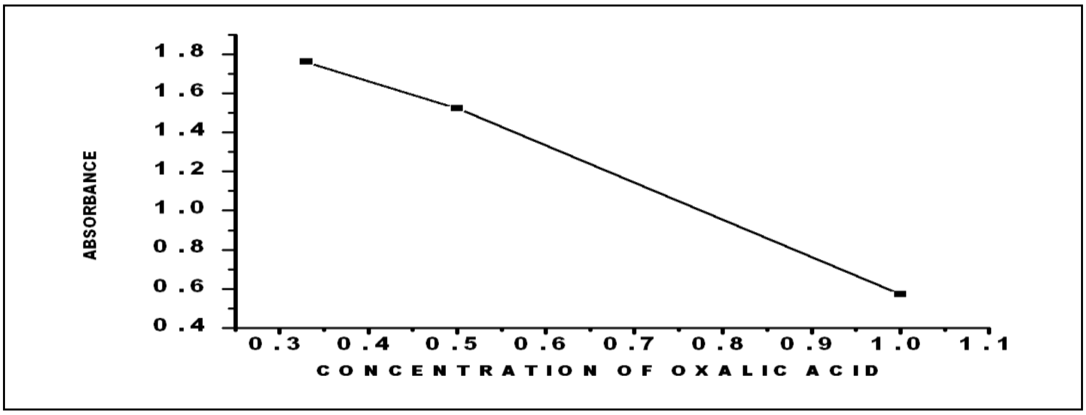

Figure6. Absorbance vs. Concentration of Oxalic acid for GOA (series)

3.3 Non linear optical studies

The first and most widely used technique for confirming the SHG from second order NLO materials is the Kurtz and Perry powder technique. The grown series of GOA crystals were illuminated with a wavelength (λ=1064 nm) of Nd:YAG laser with input enegy 1.35 mJ pulse and the corresponding second harmonic output power was recorded in terms of green light at 532 nm and collected by the photomultiplier tube. The SHG signals generated in the crystalline sample was radiation (λ=532 nm) from the crystals [14-17].It is found that as the concentration of oxalic acid increases,the conversion efficiency decreses,shown in table 3.From the study of conversion efficiency it is found that the GOA 1has maximum efficiency (Fig 7).

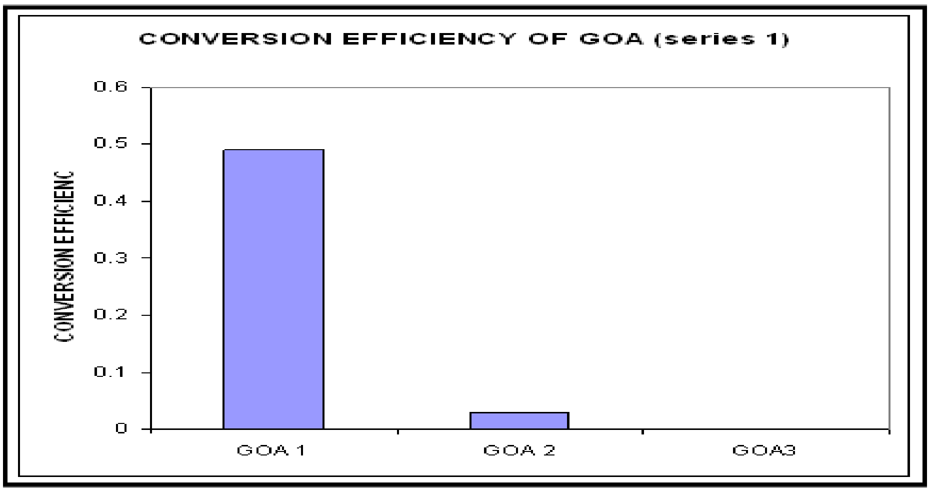

Figure 7. Conversion efficiency of GOA 1

Table3. Second Harmonic Generation conversion Efficiency of GOA (Series)

Sample	Concentration of oxalic acid	Conversion efficiency in(%)	Signal
GOA 1	0.33	0.49	27 mv
GOA 2	0.50	.03	1.7mv
GOA 3	1.0	---	0 mv
KDP	-----	---	55 mv

3.4 Photoconductivity studies

Photoconductivity is an optical phenomenon in which a material becomes more conductive due to the absorption of electromagnetic radiation such as visible light, UV light or gamma radiation. When there is no illumination a photoconductive sample has a conductance that depends on its dimension and temperature. In most cases the greater the radiant energy of a specific wavelength strikes the Surface, the higher the conductance of sample becomes up to a certain maximum. The sample shows positive as well as negative photoconductive nature. Cleaned and polished surface of the crystals have been exposed to light by using a filament lamp having iodine vapour.[18-19]. The changes in conductivity of sample in the presence and absence of light (dark current) can be recorded. The variations in photocurrent (I_P) and dark current (I_d) were determined.The enhancement of field depends on conductivity of the sample. On illumination due to absorption of photons, more of charge carriers are generated. The photoconductivity measurements were carried out on polished and silver plated samples of the grown GOA (series) samples by fixing it on microscope slide. The samples were connected in series with DC power supply and a digital picoammeter MODEL DPM111.The samples were illuminated by radiation from 100 watt lamp and photocurrent was recorded. For dark current measurement the samples were covered with a black cloth and voltage was varied in steps of 0 to 300 volts/cm. The figures 8 to 10 shows that the dark current is found to be less than the light bright current for GOA1 and GOA2 showing positive photoconductivity whereas for GOA 3 the negative photoconductivity has been recorded. As the concentration of oxalic acid decreases with respect to glycine the sample becomes negative photoconductive.

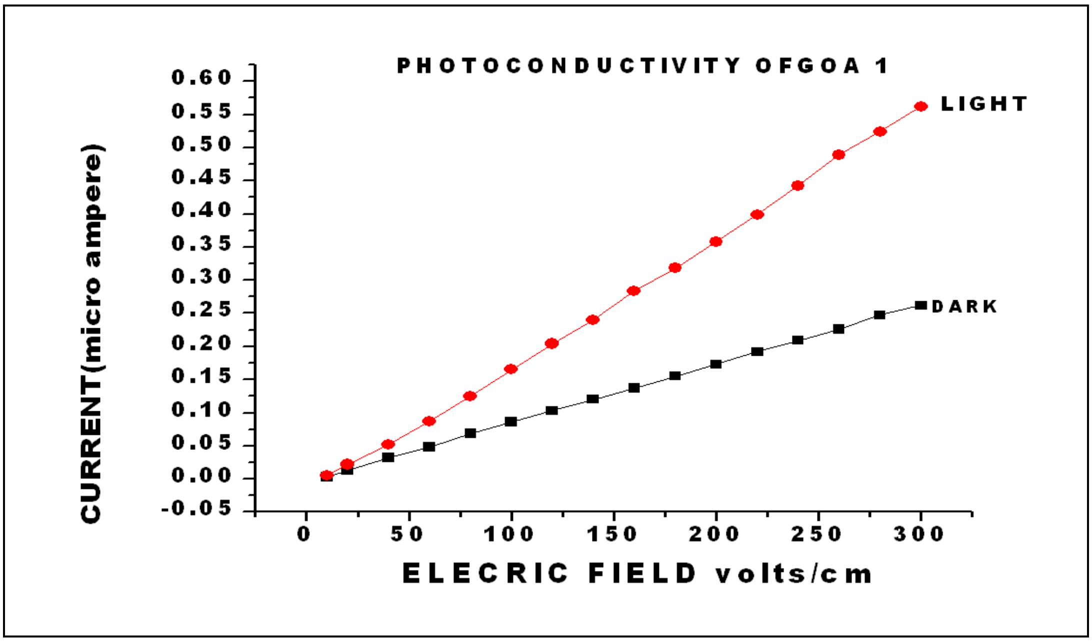

Figure8.Variation of Current with electric field forGOA1

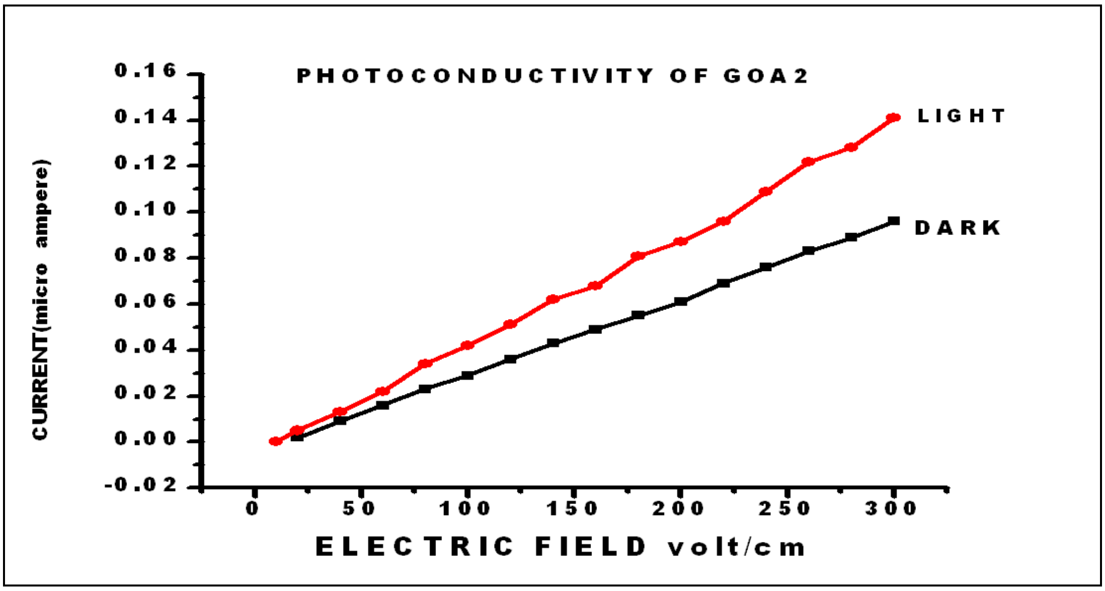

Figure9.Variation of Current with electric field forGOA2

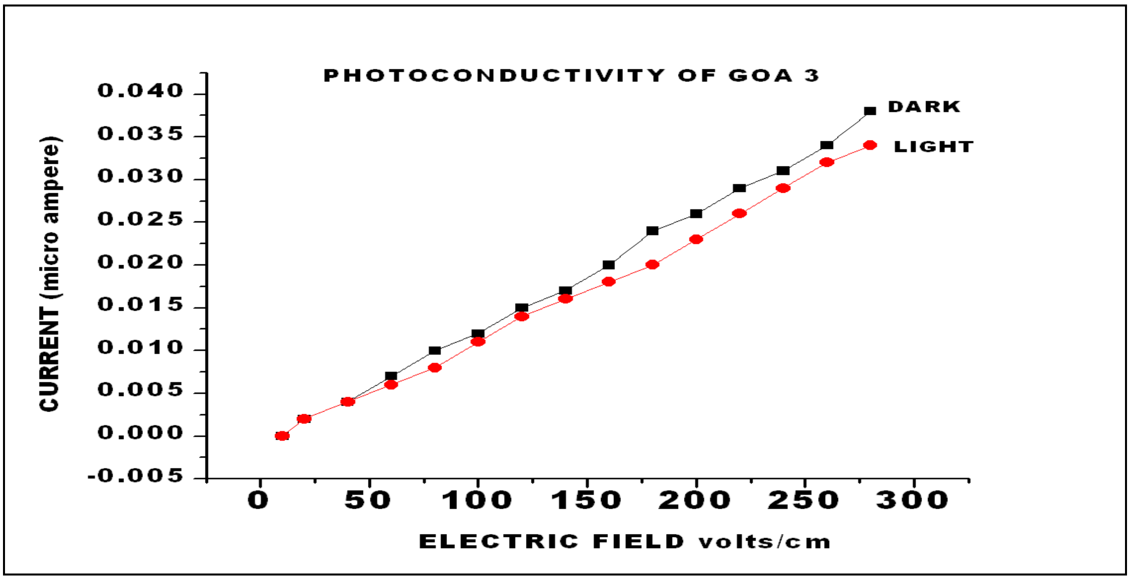

Figure10.Variation of Current with electric field forGOA3

IV. CONCLUSION

New organic GOA (glycine oxalic acid) crystals of appreciable sizes were grown from solution method in 2-3 weeks time.XRD studies reveals orhothrohmbic crystal structure. The cell parameters are dependent on the concentration of oxalic acid in the crystals. The UV visible studies shows wide transparency window between 188 nm to 700 nm suggesting the use of grown materials for non linear optical applications. The NLO studies using Nd:YAG laser shows appreciable conversion efficiency for different concentration of oxalic acid as the concentration of oxalic acid increases, the conversion efficiency decreases. The samples exhibited positive as well as negative photoconductivity at lower applied fields, with more resolution at higher applied fields>200 volts/cm, we can observe the separation between the light and dark current. As the concentration of oxalic acid decreases with respect to glycine the sample becomes negative photoconductive.

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