Journal of Applied Science and Engineering Methodologies
Volume.2, No.3, (2016): Page.282-288
www.scientistlink.com/jasem

Growth and characterization of pure and Cadmium chloride doped KDP Crystals grown by gel medium

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ABSTRACT
Crystal growth technology provides an important basis for many industrial branches. Crystals are the unrecognized pillars of modern technology. Without crystals, there is no electronic industry, no photonic industry, and no fiber optic communications. Single crystals play a major role and form the strongest base for the fast growing field of engineering, science and technology. Crystal growth is an interdisciplinary subject covering physics, chemistry, material science, chemical engineering, metallurgy, crystallography, mineralogy, etc. In past few decades, there has been a keen interest on crystal growth processes, particularly in view of the increasing demand of materials for technological applications. Optically good quality pure and metal doped KDP crystals have been grown by gel method at room temperature and their characterization have been studied. Gel method is a much uncomplicated method and can be utilized to synthesize crystals which are having low solubility. Potassium dihydrogen orthophosphate KH2PO4 (KDP) continues to be an interesting material both academically and industrially. KDP is a representative of hydrogen bonded materials which possess very good electro – optic and nonlinear optical properties in addition to interesting electrical properties. Due to this interesting properties, we made an attempt to grow pure and cadmium chloride doped KDP crystals in various concentrations (0.002, 0.004, 0.006, 0.008 and 0.010) using gel method. The grown crystals were collected after 20 days. We get crystals with good quality and shaped. The dc electrical conductivity (resistance, capacitance and dielectric constant) values were measured at frequencies in the range of 1 KHZ and 100 HZ of pure and cadmium chloride added crystal with a temperature range of 40^0C to 130^0C using simple two probe setup with Q band digital LCR meter present in our lab. The electrical conductivity increases with increase of temperature. The dielectric constants of metal doped KDP crystals were slightly decreased compared to pure KDP crystals.

Introduction:
Potassium dihydrogen orthophosphate (KDP) KH_2PO_4 continues to be an interesting material both academically and industrially. As a crystal, KDP is noted for its non-linear optical properties. KDP is used in optical modulators and for non-linear optics such as second – harmonic generation (SHG). The demand for high quality large single crystals of KDP increase due to the application as frequency conversion crystal in inertial confinement fusion [1-2]. The piezo-electric property of KDP crystal is used in the construction of crystal filters and frequency stabilizers in electronic circuit's. Also to be noted is KD*P, potassium dideuterium phosphate, with slightly different properties. Highly deuterated KDP is used in non – linear frequency conversion of laser light instead of protonated (regular) KDP due to the fact that the replacement of protons with deuterons in the crystal shifts the third overtone of the strong OH molecular stretch to longer wavelengths, moving it mostly out of the range of the fundamental line at ~1064 nm of neodymium-based lasers. Regular KDP has absorbances at this wavelength of approximately 4.7-6.3%/cm of thickness while highly deuterated KDP has absorbances of typically less than 0.8%/cm. A varying electric field applied to a medium will modulate its index of refraction. This change in the index of refraction can be used to modulate light and make it carry information. A crystal widely used for its pockels effect is potassium dihydrogen phosphate, which has good optical properties and low dielectric losses even at microwave frequencies . Many studies on the growth and properties of KDP crystals in the presence of impurities have been reported [3 - 4]. Potassium dihydrogen phosphate (KDP) crystal draws persistent attention of scientists due to its excellent quality and possibility of growing large- size crystals [5 - 6]. Microscopically, crystal growth includes crystal morphology, crystal defects, and growth rate, which are all related to the constituent growth units and their chemical bonding process [7 - 8]. KDP, ADP and DKDP are the only nonlinear crystal currently used for these applications due to their exclusive properties The grown crystals were characterized using dielectric constant, electrical properties, optical transmittance, for pure and $CdCl_2$ doped KDP crystals.

Materials and Methods:

Pure and $CdCl_2$ doped KDP single crystals are grown in sodium meta silicate gel medium using analar grade KDP and $CdCl_2$ with in concentration of 0.002, 0.004, 0.006, 0.008 and 0.010 of dopant & sodium meta silicate (1.08g/cc) .During the process pH was maintained at 5-6 at room temperature figure(1). Ethyl alcohol of equal volume is added over the set gel without damaging the cell surface .When the alcohol diffuses into the set gel, it reduces the solubility. This induces nucleation and the nuclei are grown into the single crystals. The crystal growth was carried out at room temperature. The growth period was about 20 days for pure and $CdCl_2$ doped KDP crystals. Pure and KDP doped crystals are shown in the fig (2).

Fig (1)

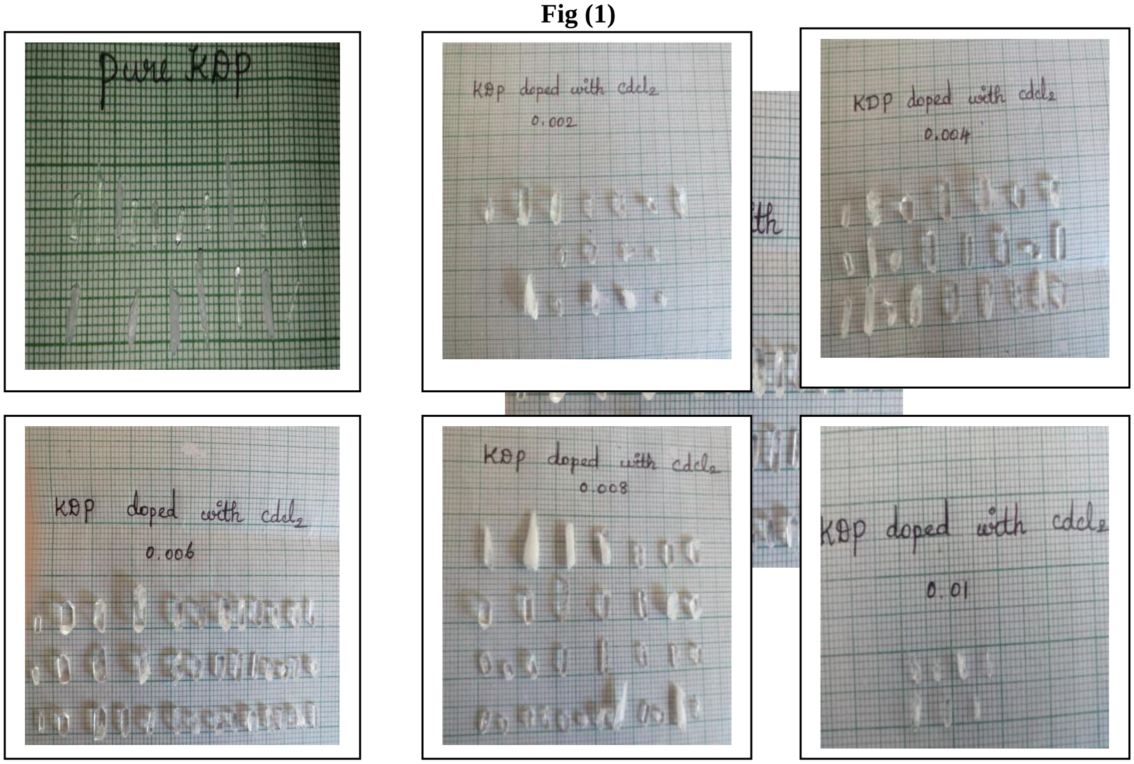


Fig (2) Pure and KDP doped Crystals

Doping:

Doping means adding impurity to the known pure crystal. To prepare a doped crystal a required amount of doping solute is also mixed along with the pure solute.

Table 1: Doping concentrations of impurities

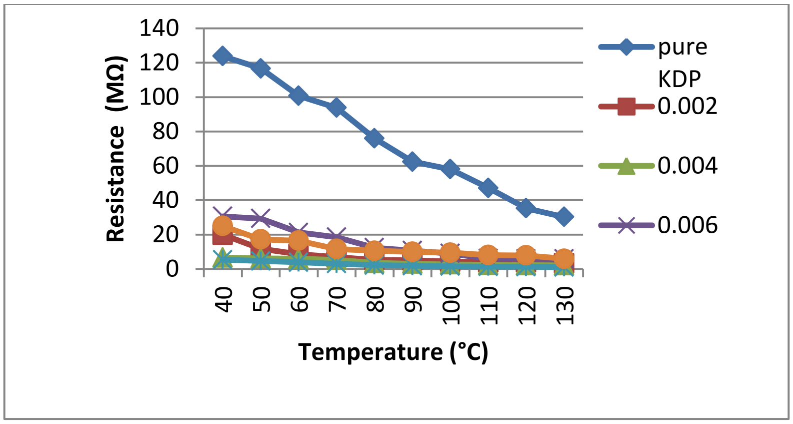
Doping ratio	**Mass of $CdCl_2$ Added**
1 : 0.002	0.05033
1 : 0.004	0.10066
1 : 0.006	0.15099
1 : 0.008	0.20132
1:0.010	0.25165

An impurity can suppress, enhance or stop the growth of crystal completely. It usually acts on certain crystallographic faces. The effects depend on the impurity concentration, super saturation, temperature and pH of the solution.

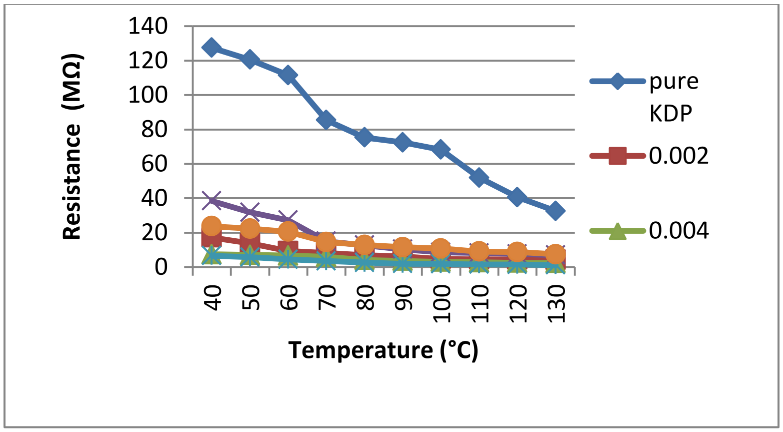
Results and Discussions:

Dielectric constant and electrical conductivity studies :

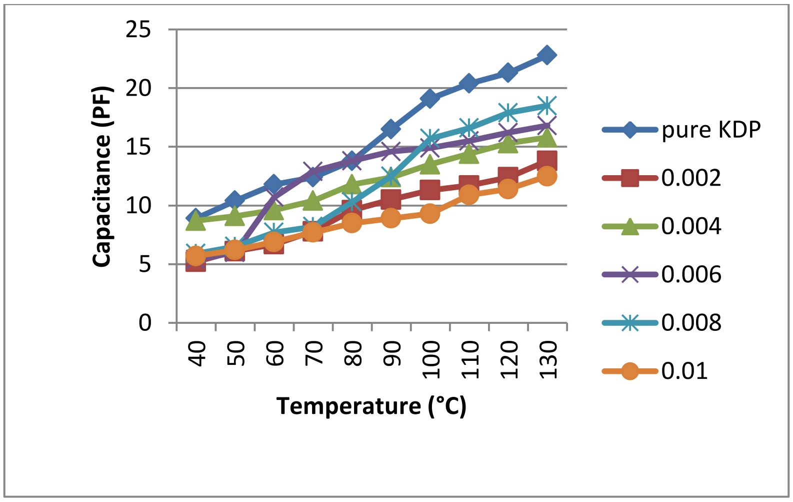
Dielectric properties are correlated with electro-optic properties of the crystals particularly when they are non-conducting materials. Due to the incorporation of metal ions polarization increases and the electrical conductivity increases. The magnitude of dielectric constant depends on the degree of polarization, charge displacement in the crystal. The dielectric constant of materials is due to the contribution of electronic, ionic, dipolar and space charge polarizations which depends on the frequencies [5]. At low frequencies, all these, polarization are active lower frequencies and high temperatures [6], in KDP crystals, many reports are available about its dielectric behavior and in our present work the measured dielectric constant values are in good agreement with the reported results [7-8]. The temperature dependence of dielectric constant at frequency 100Hz to 1KHz is shown in fig. (9 & 10). Even though KDP has many reports on dielectric loss, the study clearly ensures the crystalline perfection of crystals in our present case; it is observed that the dielectric loss decreases with increasing frequency and low dielectric losses were observed for the gel method crystal compared to the solution growth. The lower value of dielectric constant is a suitable parameter for the enhancement of SHG signals. The measurement of dielectric constant and loss as a function of frequency at different temperatures give an idea about the electrical processes that are taking place in materials and these parameters were measured on the polished (010) face of the pure and metal doped KDP crystals. Frequency dependences of dielectric constant of these crystals at room temperature were observed from the figures and it is observed that dielectric constant of KDP and $CdCl_2$ doped KDP crystals were high at low frequencies and they decreases with increase in frequency. The very high value of ϵr at low frequencies may be due to the presence of all four polarizations namely space charge, orientation, electronic and ionic and its low values at higher frequencies may be due to the loss of significance of these polarizations gradually. The nature of decrease of ϵr frequency suggests that pure and $CdCl_2$ doped KDP crystals contain dipoles of continuously varying relaxation times.



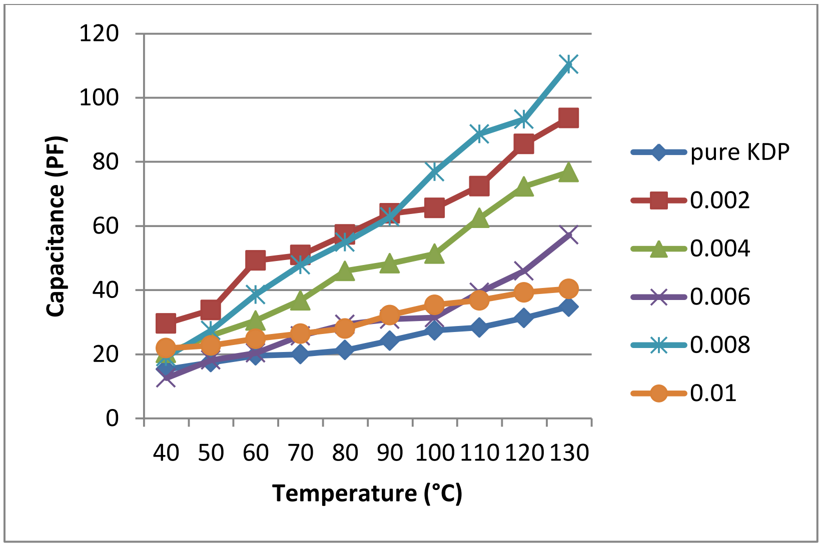
Fig(3) Variation of Resistance with temperature at frequency of 1KHz for pure and $CdCl_2$ doped KDP crystals.



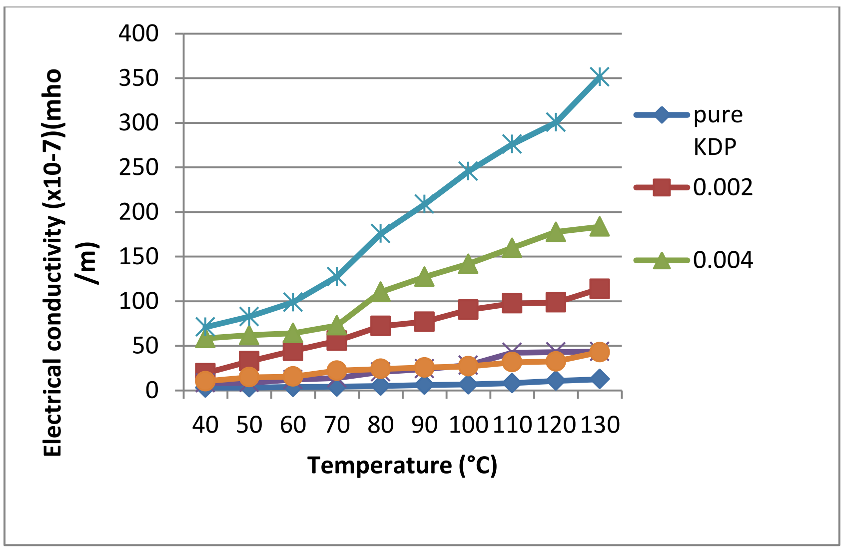
Fig(4) Variation of Resistance with temperature at frequency of 100Hz for pure and $CdCl_2$ doped KDP crystals.



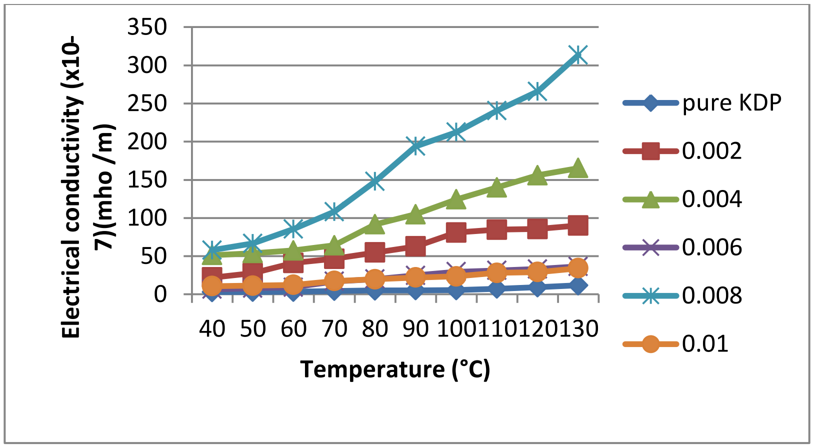
Fig(5) Variation of Capacitance with temperature at frequency of 1KHz for pure and $CdCl_2$ doped KDP crystals



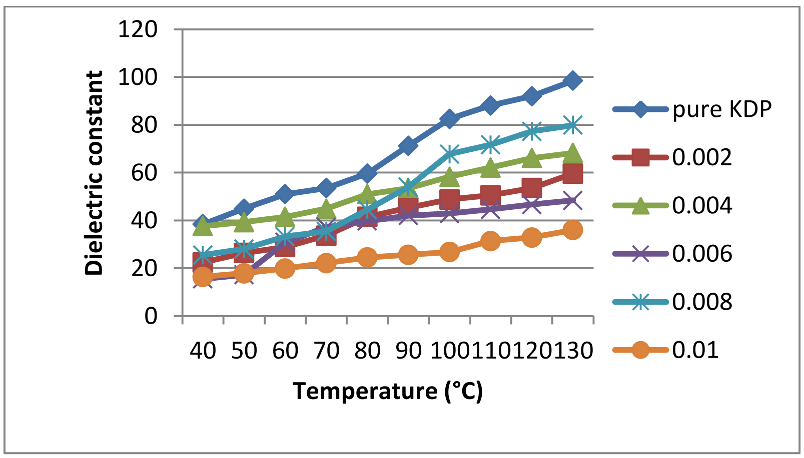
Fig(6) Variation of Capacitance with temperature at frequency of 100Hz for pure and $CdCl_2$ doped KDP crystals



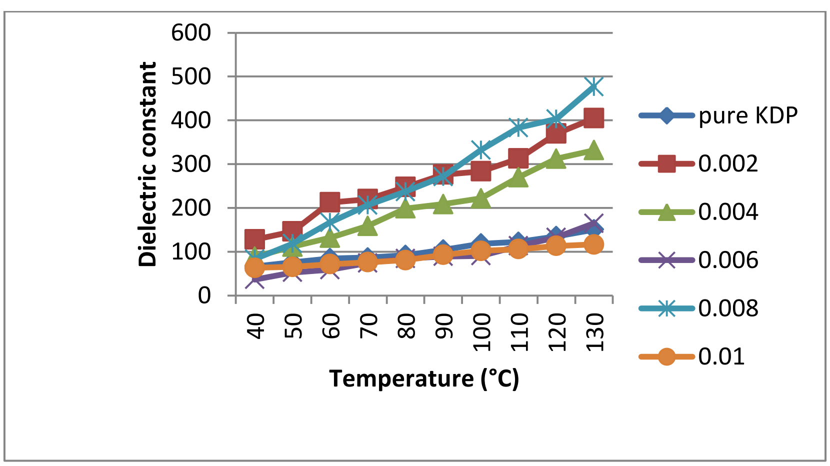
Fig(7) Variation of electrical conductivity with temperature at frequency of 1KHz for pure and $CdCl_2$ doped KDP crystals



Fig(8) Variation of electrical conductivity with temperature at frequency of 100Hz for pure and $CdCl_2$ doped KDP crystals



Fig(9) Variation of dielectric constant with temperature at frequency of 1KHz for pure and $CdCl_2$ doped KDP crystals



Fig(10) Variation of dielectric constant with temperature at frequency of 100Hz for pure and $CdCl_2$ doped KDP crystals

Conclusion

Pure KDP crystals and metal doped KDP crystals were grown by gel method. In gel growth, due to the three dimensional structures, the crystals were free from microbes. The resistance, capacitance, dielectric constant and electrical conductivity were measured at frequencies in the range of 1KHz and 100Hz of pure and $CdCl_2$ doped KDP crystals. The capacitance and dielectric constants of metal doped KDP crystals were slightly decreased compared to pure KDP crystals. The lower the value of dielectric constant more is the enhancement of SHG signals. The electrical conductivity of the pure KDP and $CdCl_2$ doped KDP crystals were found to be increase with increase of temperature and frequencies.

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