RAPID CRYSTAL GROWTH OF BENZOPHENONE BY LOW TEMPERATURE SOLUTION GROWTH AND ITS CHARACTERIATION

Dr.I.Cicilignatius1.,Dr.K.Palanisamy2.,Dr.Balachandran3.,Ms.P.Dhivya4.
Assistant Professor Department of Physics
DhanalakshmiSrinivasan College of Arts & Science for Women

ABSTRACT

Low temperature resolution growth (LTSG) is one in every of the foremost economical and simplest processes which might be used for crystal growth from solutions. the convenience in handling and also the readiness in its miscibility with the solvents create it a sexy technique for crystal growth. Most of the chosen solutes are compatible with water and a few ar2 with alcohol and ethers. Benzophenone a photoinitiator employed in the UV-curing applications in inks, adhesive and coatings, optical fibers and in computer circuit boards. Benzophenone or di-phenyl organic compound (CAS No. 119-61-9) with formula (C6H5)2CO is compatible with alcohol in best proportions. the answer is heated for supersaturation well below its boiling purpose. The saturated resolution is then takenin an exceedingly beaker and unbroken for crystallization. traditional nucleation followed by crystallization happens by evaporation technique at temperature. A provision for speedy evaporation by a stream of uniform hot air flow across the expansion chamber enhances the crystallization rate thereby reducing the time needed for the expansion of crystals. This acceleration of crystal growth is a crucial necessity for the mass crystallization and industrial crystal growth. The crystals are analysed for his or her surface, structural and spectroscopical characteristics.

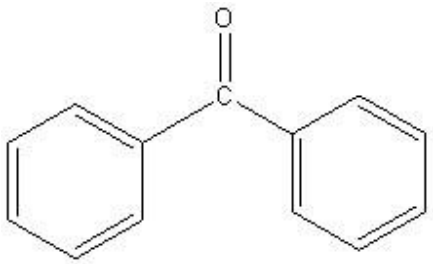
Keywords:

Benzophenone, photoinitiator, ultra-violet filter, Nonlinear optic, Absorption, Transmission, FTIR.

Introduction:

Benzophenone, associate degree aromatic organic compound (diphenyl ketone), is a crucial compound in organic chemistry and perfumery yet as in organic synthesis. it's a white crystalline substance with rose-like odor; insolube in water; purpose|freezing point|temperature} temperature boiling point 305 - 306 C. Benzophenone is employed as a constituent of artificial perfumes and as a beginning material for the manufacture of dyes, pesticides and medicines (especially anxiolytic and hypnotic drugs). Benzophenone is employed as a photoinitiator of UV-curing applications in inks, adhesive and coatings, fibre yet as in computer circuit boards. Photoinitiators ar compounds that break down into free radicals upon exposure to ultraviolet. Photoinitiators bear a unimolecular bond cleavage upon irradiation to yieldfree radicals (benzoin esters; benzil ketals; alpha-dialkoxy acetophenones; alpha- group alkylphenones; alpha-amino alkylphosphine; acylphosphine oxides). Another variety of photoinitiators bear a unit reaction wherever the excited state of the photoinitiator interacts with a second molecule (a co-initiator) to get free radicals (benzophenones, amines; thioxanthones, titanocenes). ultraviolet has additional energy than visible

radiation, and so degrade the physical properties such because the look of organic substances and plastics. Benzophenones will act as optical filters or deactivate substrate molecules that are excited by light-weight for the protection polymers and organicsubstances. Their, cosmetic grades, ar used as cream agents to cut back skin injury by obstruction actinic ray. Benzophenone could be a promising NLO material wherever the absorption is visible within the blue light-weight region having a cut-off wavelength below 450nm [1,2,3].



benzophenone
$C_{13}H_{10}O$

Properties of Benzophenone :

Molecular formula : C_{13} H_{10} O

Molar mass : 182.22 g. mol^{-1}

Appearance : White solid

Odor : Germanium[1]

Density : 1.11 g/cm^{3}[1]

Melting point : 48.5ºC (119.3ºF; 321.6 K)[1]

Boiling point : 305.4ºC (581.7º F; 578.5 K)[1]

Solubility in water : Insoluble[1]

Solubility in organic solvents : 1 g/7.5 mL in ethanol[1]

Organic solvent : 1 g/6 mL in diethyl ether[1]

Uses :

Benzophenon will be used as a photograph instigator in UV - falcate application[2] like inks, imaging, and clear coatings within the printing business. Benzophenon prevents ultraviolet (UV) light-weight from damaging scents and colours in product like perfumes and soaps. Benzophenon may be extra to plastic packaging as a UV blocker to stop ikon - degradation of the packaging polymers or its content. Its use permits manufactures to package the merchandise in clear glass or plastic (such as a PETE water bottle)[3]. without it, opaque or dark packing would be needed. In biological applications, Benzophenon are used extensively as photophysical probes to spot and map amide - supermolecule interactions.

Substrate and experimental technique :

Growth of crystals from binary compound solutions is one in every of the traditional ways of crystal growth. the strategy of crystal growth from coldbinary compound solutions is extraordinarily well-liked within the production of the many technologically necessary crystals. it's the foremost wide used technique for the expansion of single crystals. the expansion of crystals by cold resolution growth involves weeks, months and generally years. although the technology of growth of crystals from resolution has been well formed, it involves meticulous work, a lot of patience and even a touch quantity of luck. an influence failure or a contaminated batch of stuff will destroy months of labor.

Solid salt of Benzophenon is taken and dissolved in amount of alcohol till it reaches saturation state. The material is heated by order to obtain the supersaturated state which the salt becomes unsoluble in the solvent like alcohol. The solubility and supersolubility of Benzophenone was observed to be 5 grams and 7.5 grams per 100ml of ethanol solvent. Then the supersaturated solution is then filtered using Filter paper (pore size 5 micron) and left inside the constant temperature solution growth process for nucleation and subsequent crystallization. The nucleation time was measured for repeatability and was observed to be 226seconds.

The solution was left for crystallization in a constant temperature water bath where the ambient temperature of the bath was optimally kept at 35˚C for complete solute utilization where the solvent was found to evaporate fully forming Benzophenone Crystals in the beaker in which it was kept togrow.

As grown crystals of Benzophenone were cleaved and presented for observation. A cleaved sample of size 2.25 cm x 2.25 cm x 2 cm was the largest crystal out of the sample rapidly crystallized using solution growth apparatus.

Microindentation Tests

Microindentation hardness testing (MHT) is a very valuable tool for the materials engineer, but it must be used with care and a full understanding of potential problems. The purpose of MHT is to study fine scale changes in hardness, either intentional or accidental. The most common microindentation tests are the Vickers and the Knoop tests. In the Vickers test, the load is applied smoothly without impact and held in place for 10 to 15 seconds. After the load is

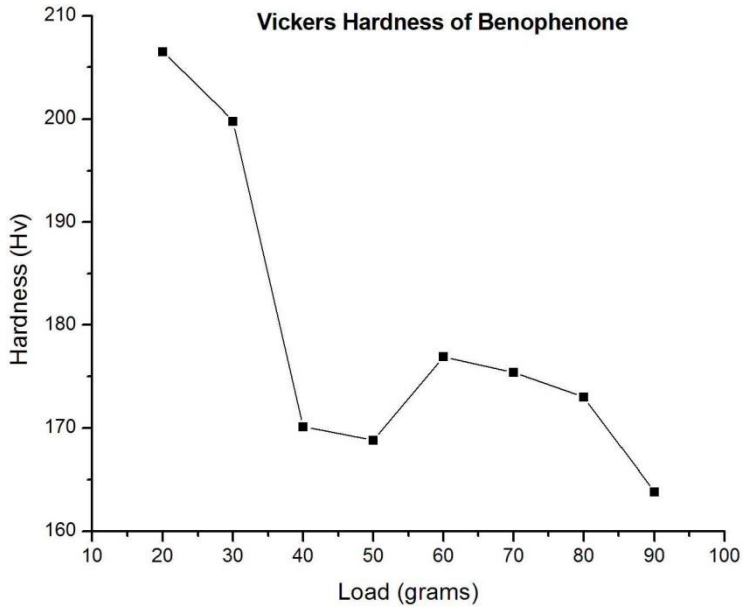
removed, the two impression diagonals are measured and then averaged. The Knoop test is conducted in the same manner as the Vickers test. However, only the long diagonal is measured, except for the projected area hardness test. The factors affecting the precision and bias in MHT are discussed.

Among the various method of hardness measurements, the most common and reliable method is the Vicker's hardness test method. It was done using a Leitz Wetzlar Microhardness tester and observed using a Metallux microscope. In this method, microindentation is made on the surface with the help of a diamond pyramidal indentor. Smith and Sandland have proposed that a pyramid be substituted for a ball in order to provide geometrical similitude under different values of load. The Vicker's pyramid indentor where opposite faces contain an angle (=136) is the most widely accepted pyramid indentor. Pyramid indentors are said to be best suited for hardness tests due to two reasonsnamely

i).Thecontactpressureforapyramidindentorisindependentofindentsize.
ii).Pyramidindentersarelessaffectedbyelasticreleasethanotherindentors.

iii).The base of the Vicker's pyramid is a square and the depth of indentation corresponds to $1/7^{th}$ of the indentation diagonal.

Benzophenone crystal had a maximum Vickers microhardness value of 206 for 20grams and a minimum of 162 for 90 gramsload.



XRD-ANALYSIS

Powder X-Ray diffraction was carried out with the as grown crystals in powdered form. The powdered samples were loaded into a Rigaku X-ray

diffraction apparatus using CuKα radiations having λ=1.5045 Å and analysed for confirmation. The crystallographic data of the lattice parameters [4] obtained were a=9.751Å, b=10.671 Å, c=24.813 Å, α=87.47, β=83.80, γ=63.154 . Benzophenone exhibited as a face centered monoclinic structure with space group C and volume of the unit cell was V=2290.46 Å3. The results were compared with the ICDD database files and conformed with four of the available peak values (200), (220), (103) and(321)

Fig.5: Powder XRD of Benzophenone

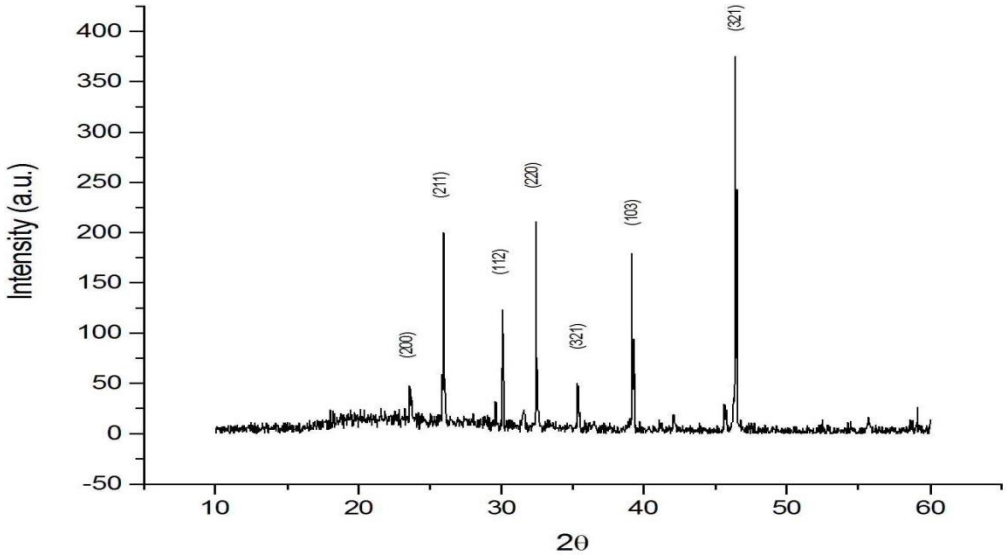
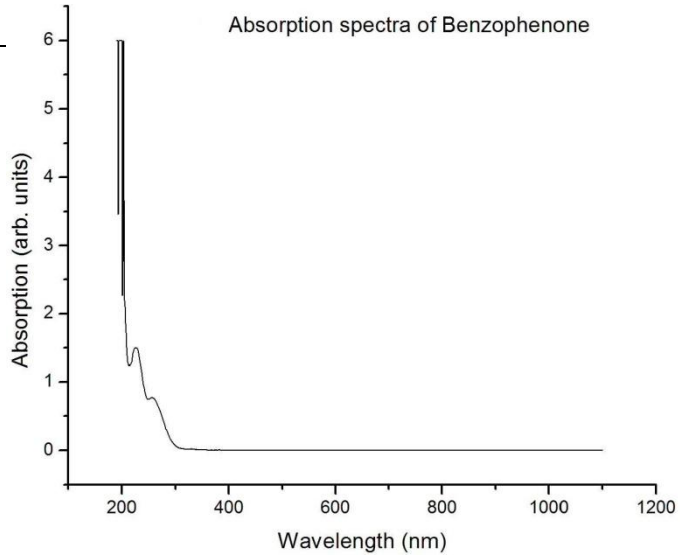


Fig.6: UV-VIS Absorption Spectra of Benzophenone



CONCLUSION

Crystals of Benzophenone were grown rapidly by low temperature solution growth technique at room temperature. The structural, optical and qualitative NLO effeciency properties were analysed. The microhardness values were found to be higher than reported values [5]. The UV-VIS spectra recorded a near 95% transparency for the cleaved samples of Benzophenone along the (001) orientation. The FTIR analysis proves that the assignments were in order for Benzophenone depicting aromatic consistency and confirmatory in individual band assignments.

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