

# INVESTIGATIONS AND SYNTHESIS OF SILVER-DOPED PHTHALIC ANHYDRIDE SINGLE CRYSTAL AND ITS EFFECT ON ANTIBACTERIAL ACTIVITY

G. Jebadoss Thinakaran, R. Sugaraj Samuel<sup>✉</sup>, M. Ajith Kumar  
and J. Prince Joshua

Department of Physics, The New College, Chennai-600014, India  
(Affiliated to the University of Madras)

<sup>✉</sup>Corresponding Author: [sugarajsamuel@yahoo.com](mailto:sugarajsamuel@yahoo.com)

## ABSTRACT

The objective of this study was to introduce phthalic anhydride into Silver, to produce a novel substance having biological activity. The Fourier Transform Infrared Spectroscopy (FTIR), Powder X-ray Diffraction (PXRD), Ultraviolet-Visible Spectroscopy (UV-visible), and antimicrobial analyses collectively yielded compelling evidence indicating the occurrence of chemical alteration in the crystal structure. The obtained materials had a remarkable inhibitory action against *K. pneumoniae* and *S. aureus*, resulting in almost complete suppression. Additionally, they also hindered the growth of *S. epidermidis*. The derivatives exhibited the highest level of antibacterial activity, indicating that the addition of phthalic groups resulted in the hydrophobization of Silver, thereby improving the interaction between these compounds and the constituents of the bacterial cell wall. The Fourier Transform Infrared (FTIR) analysis yields data about the functional groups present in a sample, whilst the Ultraviolet (UV) examination provides insights into its optical properties. The present study presents a novel approach to introduce dopants into single crystals of phthalic anhydride, so greatly enhancing their potential for environmental applications.

**Keywords:** Phthalic Anhydride, Single Crystals, XRD, FTIR, UV-Visible, Antibacterial.

RASAYAN J. Chem., Vol. 17, No.1, 2024

## INTRODUCTION

The significant interest in nonlinear optical (NLO) materials stems from their potential applications across several fields, like medical diagnosis, underwater communication, telecommunications, high optical data storage, and color display systems.<sup>1,2</sup> The utilization of various application-oriented single crystals has played a pivotal role in enabling the advancement of novel device creation within the realm of technological society. According to the literature, organic crystals exhibit significant nonlinearity, but they possess relatively low mechanical strength and thermal stability. On the other hand, inorganic materials demonstrate moderate nonlinear behavior along with better mechanical and thermal stabilities.<sup>3</sup> There is a category of materials that combines the nonlinear properties of organic materials with the thermal stabilities and mechanical of inorganic materials, aiming to overcome the limitations associated with both organic and inorganic materials. The user has provided a numerical range of.<sup>4,5</sup> The multifaceted utility of organic crystals in the fields of superconductivity, semiconductor technology, and photonics is widely acknowledged. The organic nonlinear optical (NLO) materials are constructed using an extended  $\pi$ -bond system that spans a significant portion of the molecule. This system can be easily altered by introducing electron-giving and electron-withdrawing groups in proximity to aromatic moieties. Consequently, this modification leads to an augmentation in optical nonlinearity. The limited presence of widespread  $n$ -electron delocalization in inorganic crystals results in their relatively modest nonlinear susceptibilities. The emergence of semi-organic materials can be attributed to the ongoing pursuit of efficient nonlinear optical (NLO) materials for contemporary technology.<sup>6</sup> These materials provide a unique combination of the strong mechanical properties seen in inorganic materials and the high nonlinear susceptibility characteristic of organic materials. As a result, they are widely regarded as holding great potential for advancements in the field of nonlinear optics. The introduction of a small quantity of dopant impurity significantly modifies the characteristics of crystals. A dopant has the potential to either enhance or inhibit the development rate of a crystal. The increasing prevalence of laser diodes has led to a growing interest in metal ion-doped crystals.

The addition of minute amounts of transition metals has been found to augment the pace of crystal formation, improve optical transparency, and enhance mechanical strength. In the past few years, researchers have made noteworthy advancements in the discovery of materials that possess beneficial nonlinear optical properties. These discoveries have been made possible through the utilization of specific strategies and the application of ideas related to molecular and crystal engineering.<sup>7</sup> Producing big crystals with great optical quality poses a challenge in the context of device applications. Phthalic Anhydride (PA) doped with Silver is an organic chemical that exhibits noteworthy nonlinear optical (NLO) capabilities. Fluorescence, phosphorescence<sup>8</sup>, dendritic<sup>9</sup>, and spherulitic<sup>10</sup> represent some of the intriguing characteristics exhibited by Silver-doped PA. In this study, the production of single crystals of Silver-doped PA was achieved through the implementation of a slow evaporation approach. The confirmation of the generated crystals was achieved through the utilization of various analytical techniques, like powder X-ray diffraction (XRD), UV-visible spectroscopy, Fourier-transform infrared spectroscopy (FT-IR), photoluminescence analysis, and antimicrobial testing.

## EXPERIMENTAL

A monocrystalline specimen of phthalic anhydride doped with Silver was synthesized through the dissolution of Silver nitrate and phthalic acid in methanol, employing a stoichiometric ratio of 0.2:1. The resultant mixture was stirred well for 30 minutes using a magnetic stirrer. Subsequently, the aforementioned solutions were amalgamated, subjected to rigorous agitation for 60 minutes, and subsequently underwent filtration. Subsequently, the filtrate was subjected to ambient drying conditions, leading to the formation of crystals by a method characterized by progressive evaporation. The purity of the synthesized crystal was improved by employing a sequence of recrystallization procedures, leading to the generation of high-quality, transparent single crystals within 30 days.

## RESULTS AND DISCUSSION

### Powder XRD Studies

Powder X-ray diffraction (XRD) is a valuable method employed in the verification of the composition of a solid material, as well as in the assessment of its crystalline nature and the extent to which it is free from impurities in terms of its phase. Consequently, the X-ray diffraction (XRD) pattern of the produced material was acquired. The occurrence of peaks observed in the X-ray diffraction (XRD) pattern indicates that the crystals generated possess a crystalline structure. The temperature range of 1060 °C was scanned at a rate of 1 °C per minute for the powdered material of a Silver-doped PA crystal. PROZSKI software was used for indexing the peaks.

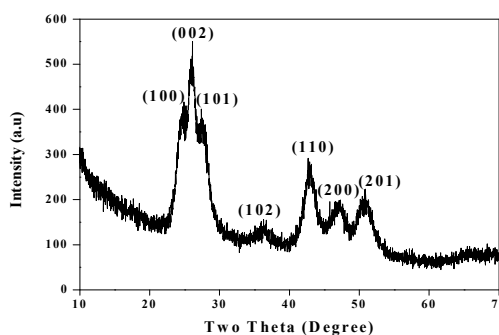


Fig.-1: P-XRD Spectra of Silver-doped phthalic anhydride (PA) Single Crystal

### FT-IR studies

The Fourier Transform Infrared (FTIR) spectrum provides additional information regarding the structural characteristics of a molecule. In this methodology, a significant majority of the functional groups present in the molecule exhibit absorption within a distinct spectrum of frequencies. The molecular bands undergo stretching and bending motions about each other due to the absorption of infrared (IR) radiation. The Fourier-transform infrared (FT-IR) spectra of Silver-doped phthalic anhydride have distinct peaks at 487.99 and 613.36  $\text{cm}^{-1}$ , which can be attributed to the symmetric and asymmetric vibrations of stretching, respectively, of the  $=\text{C}-\text{O}-\text{C}=$  linkage (ether) present in the chemical. The observed signal at 1386.81  $\text{cm}^{-1}$

can be attributed to the existence of a carbonyl group. The prominent peak observed at a wavenumber of  $3751.34\text{ cm}^{-1}$  is ascribed to the existence of the anhydride functional group.

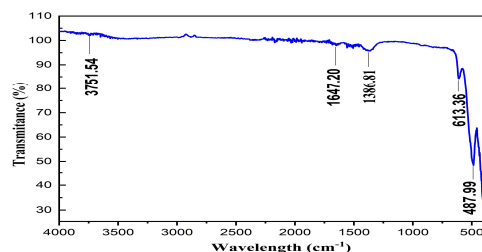


Fig.-2: FT-IR Spectra of Silver-doped phthalic anhydride (PA) Single-Crystal

### UV-Visible Studies

The absorption spectrum in the UV-visible range of a crystal can provide valuable insights into its crystal quality, encompassing the identification of faults, impurities, and lattice distortions. Variations in crystal structure or composition can be inferred from alterations in the absorption peaks or the emergence of additional absorption bands. The UV-visible spectra of methanol (solvent) containing Silver-doped PA exhibits a prominent absorption peak at approximately 250 nm. This refers to the  $\pi \rightarrow \pi^*$  transition of an aromatic ring.

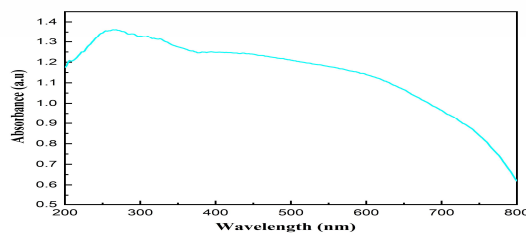


Fig.-3: UV Visible Spectra of Silver-doped phthalic anhydride (PA) Single Crystal

### Antibacterial Studies

The existent study is aimed to assess the antibacterial efficacy of the Silver-doped phthalic anhydride compound against three bacterial strains, including *Klebsiella pneumoniae*, *Staphylococcus epidermis*, and *Staphylococcus aureus*. The twofold micro-dilution method was employed to investigate the minimum inhibitory concentration (MIC) of the chemical and pristine antibiotics, namely *cefotaxime* and *ceftriaxone*. The Silver-doped phthalic anhydride composite exhibited a comparable minimum inhibitory concentration (MIC) to that of *cefotaxime* against all three bacterial strains.

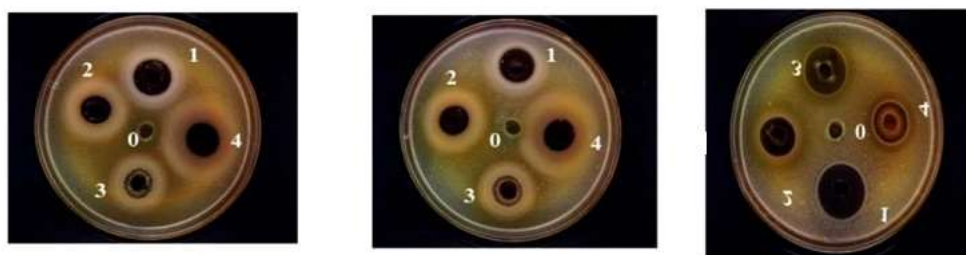


Fig.-4: Antibacterial Study of Silver-doped phthalic anhydride (PA) Single Crystal

The observed activity against *K. pneumoniae* at a dose of 2 mg/mL showed a higher efficacy compared to *ceftriaxone* (3 mg/mL). The minimum inhibitory concentration (MIC) for *S. epidermis* and *S. aureus* was found to be slightly lower at 3 mg/mL, indicating a relatively weaker effect compared to *ceftriaxone*. However, in the case of *K. pneumoniae*, the MIC of *ceftriaxone* was higher at 5 mg/mL, suggesting a lesser inhibitory effect.

### CONCLUSION

The improved precursor technique was developed, delineated, and experimentally evaluated for the synthesis of doped crystalline Silver minerals. The indexing of PXRD peaks was performed using

PROZSKI software. The UV-visible spectrum obtained in a methanol solution exhibited a pronounced absorption peak at a wavelength of 250 nm. The existence of a carbonyl group is indicated by a peak observed at  $1386.81\text{ cm}^{-1}$  in the FTIR spectrum. Additionally, the presence of an anhydride group is confirmed by the presence of a strong band noticed at  $3751.34\text{ cm}^{-1}$  in the same spectrum. The improved precursor technique was specifically devised to achieve the synthesis of Silver with the essential characteristics required for an optimal photocatalyst, including a substantial pore volume, extensive surface area, superior crystallinity, and a narrow band gap. The observed activity against *K. Pneumoniae* was notably high at a dosage of 2 mg/ml. The observed outcome exhibited superior efficacy compared to ceftriaxone at a concentration of 3 mg/mL. The minimum inhibitory concentration (MIC) for *S. epidermis* and *S. aureus* was somewhat lower at 3 mg/mL, indicating a relatively weaker effect compared to ceftriaxone. However, in the case of *k. pneumoniae*, the MIC of ceftriaxone was higher at 5 mg/mL, suggesting a less effective inhibition.

### ACKNOWLEDGMENTS

The authors acknowledge the help rendered by Dr. L. Allwin Joseph, Department of Physics, The New College, Chennai in spectral analysis and interpretation.

### CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

### AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID IDs, given below:

G. Jebadoss Thinakaran  <https://orcid.org/0009-0006-4538-3641>

R. Sugaraj Samuel  <http://orchid.org/0000-0002-2212-1052>

J. Prince Joshua  <http://orchid.org/0000-0002-2818-9111>

M. Ajith Kumar  <https://orcid.org/0009-0006-4538-3641>

**Open Access:** This article is distributed under the terms of the Creative Commons Attribution 4.0 International License (<http://creativecommons.org/licenses/by/4.0/>), which permits unrestricted use, distribution, and reproduction in any medium, provided you give appropriate credit to the original author(s) and the source, provide a link to the Creative Commons license, and indicate if changes were made.

### REFERENCES

1. S. Janarthanan, Y. C. Rajan, R. Sugaraj Samuel, S. Pandi, *Advanced Materials Research*, **584**, 140(2012), <https://doi.org/10.4028/www.scientific.net/AMR.584.136>
2. G. Centi, *Catalysis Today*, **16(1)**, 05(1993), [https://doi.org/10.1016/0920-5861\(93\)85002-H](https://doi.org/10.1016/0920-5861(93)85002-H)
3. K. Maheshaa Upadhya and N. K. Udayashankar, *Synthesis and Reactivity in Inorganic Metal-Organic and Nano-Metal Chemistry*, **40(10)**, 820(2010), <http://doi.org/10.1080/15533174.2010.522548>
4. F. Cavani, A. Colombo, F. Trifiro, M.T. Sananes Schulz, J.C. Volta and G.J. Hutchings, *Catalysis Letters*, **43**, 267(1997), <http://doi.org/10.1023/A:1018927714568>
5. F. Ben Abdelouahab, R. Olier, N. Guilhaume, F. Lefebvre and J.C. Volta, *Journal of Catalysis*, **134**, 167(1992), [https://doi.org/10.1016/0021-9517\(92\)90218-7](https://doi.org/10.1016/0021-9517(92)90218-7)
6. K. Mahesha Upadhya and N.K. Udayashankar, *Synthesis, Microhardness*, *International Journal of Thermophysics*, **33**, 142(2012), <https://doi.org/10.1007/s10765-011-1134-4>
7. S. Albonetti, F. Cavani, F. Trifirò, P. Venturoli, G. Calestani, M. Lopez Granados and J.L.G. Fierro, *Journal of Catalysis*, **160(1)**, 64(1996), <https://doi.org/10.1006/jcat.1996.0123>
8. Zhang-Lin.Y, Forissier.M, Vedrine, J.C. and Volta. J.C., *Journal of Catalysis*, **145(2)**, 275(1994), <https://doi.org/10.1006/jcat.1994.1034>
9. G.A. Sola, B.T. Pierini and J.O. Petunchi, *Catalysis Today*, **15(3,4)**, 545(1992), [https://doi.org/10.1016/0920-5861\(92\)85018-H](https://doi.org/10.1016/0920-5861(92)85018-H)
10. J. George and S. K. Premachandran, *Journal of Physics D: Applied Physics*, **12(7)**, 1129(1979), <https://doi.org/10.1088/0022-3727/12/7/017>

[RJC- 8673/2023]