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Data Article

Data on *in-vitro* antibacterial activity and third order NLO property of 2-aminopyridine copper sulphate (2APCS)

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ABSTRACT

Solution growth technique was used to harvest nonlinear optical 2-aminopyridine copper sulphate (2APCS) crystal at room temperature. Crystallinity in 2APCS crystal was confirmed by powder XRD. Single XRD study reveals 2APCS crystallizes in triclinic system. Band assignments of synthesized compound were identified using FT-IR study. Linear optical study shows lower cut-off wavelength 237 nm for 2APCS. Mechanical behavior study of 2APCS crystal posses soft nature of the sample. Third harmonic generation (THG) study was analyzed using Z-scan Technique for 2-aminopyridine copper sulphate (2APCS) crystal. *In-vitro* antibacterial test was carriedout for the 2APCS sample using test pathogenic bacteria, Escherichia coli (MTCC 443), S.aureus (MTCC 96) and E.faecalis (MTCC 439) were tested to find the antimicrobial activity.

Specifications Table

Subject area	Semiorganic materials, crystallographic, nonlinear optics and Pharmaceuticals
Compounds	2-Aminopyridine ($C_5H_6N_2$) and Copper sulphate ($Cu\ So_4$)
Data Category	Crystallographic, Nonlinear Optical and Anti-microbial
Data acquisition format	Data analysis and processing
data type	analyzed data
Procedure	Slow evaporation solution growth technique, room temperature and well diffusion method
Data accessibility	Data is provided on request

1. Rationale

A semiorganic nonlinear optical crystal attests various applications in the field of opto-electronics, optical switching, frequency conversion, telecommunications and device fabrications [1]. Semiorganic material have combined properties with organic and inorganic to develop nonlinear optical application in device fabrications [2]. When organic material combines with metal complexes, it relent non-centrosymmetric system by suitability of NLO property [3, 4].

Organic centrosymmetric structure changed into a noncentrosymmetric one by facilitating metal complexes into that as a semiorganic using various physicochemical strategies and their potential usability in NLO devices as a reliable frequency conversion one having large birefringence, sample transparency, high laser harm threshold with chemical and mechanical stability have been reported [5, 6].

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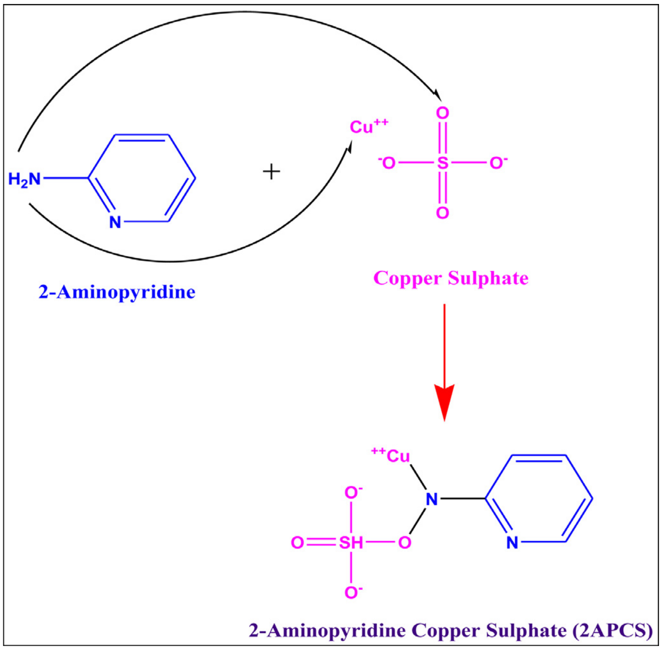

Fig. 1. Scheme of 2APCS.

Especially, third order nonlinear optical (TONLO) materials are very fascinated in the field of optics because of their new modes of action display in ultrafast optical switching/limiting and photonic devices [7–10]. In recent literature, carboxylic groups as striking substrate for fabrication due to capability in supramolecular modes interaction of hydrogen among themselves [11, 12]. Pyridinium complexes play an important role to constitute the NLO materials with charge transfer system and donor–acceptor properties, and enable the changes in physical and structural properties [13]. pyridine derivatives show a specific aspect in pharmaceutical applications due to their heterocyclic nature [14–16]. 2-aminopyridine ligands as ocular materials [17–20] and its suitability for optical device fabrications are reported [21, 22].

Copper compounds, have vital applications in biological, perpetuation, photoluminoscent behavior, enzyme reaction, mesmeric reactions, resistive element in liquid resistors and mainly used as a fungicide and herbicide [23–26]. Prominent aspect in the progress of physics makes probable important due to their microscopic properties. In this research article, copper binding with 2-aminopyridine to get better ocular and biological properties. The various characterization techniques are carried-out and their results are detailed.

2. Experimental procedure

2-aminopyridine copper sulphate (2APCS) salt was synthesized using de-ionized water as a solvent by taking AR grade of 2-aminopyridine and copper sulphate in equimolar ratio 1:1. This synthesized solution was stirrer well for 6 h using magnetic stirrer to ensure homogeneity in the aqueous solution of 2APCS at ambient temperature. Scheme of the synthesized 2APCS has been given in the following chemical reaction shown in Fig. 1.

A prepared solution of 2-aminopyridine copper sulphate (2APCS) was successfully recrystallized to form homogeneity in growth process. Using magnetic stirrer the solution was stirred continuously about 9 h till high purity of synthesized compound was achieved. The solution was filtered thrice using Whattman filter paper and the filtered solution was poured into a beaker of 500 ml and covered with porous paper for slow evaporation process. The whole setup was kept in dust free area and closely monitored. Small crystals appeared in the beginning of 25 days, due to evaporation a good optical crystal in dimension $08\times05\times03$ mm^3 were harvested in the period of 85 days. As grown 2APCS crystal was shown in Fig. 2.

3. Values of the data

A powder sample of 2APCS was subjected to powder X-ray diffraction analysis using Rich Seifert Powder X-ray diffractometer with K_α radiation (λ=1.5406 Å). The sample 2APCS was scanned in the range between 10° to 80°. The powder indexed diffractometer of 2APCS crystal was shown in Fig. 3. The sharp intensity peaks found in XRD pattern hows good crystalline nature and purity of the grown crystal.

Single XRD analysis is most often used to determine the internal lattice of crystalline substance of materials. A good quality crystal was selected for XRD studies. The unit cell parameter values are a = 8.84 Å; b = 10.39 Å; c = 16.14 Å;

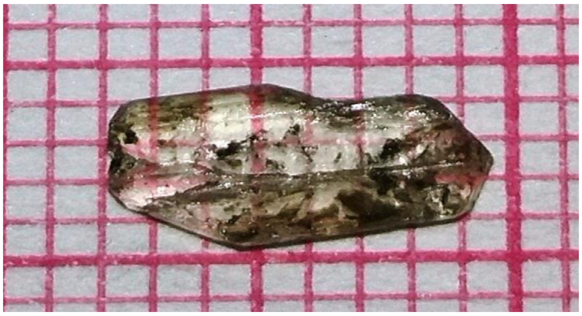

Fig. 2. Photography of 2APCS crystal.

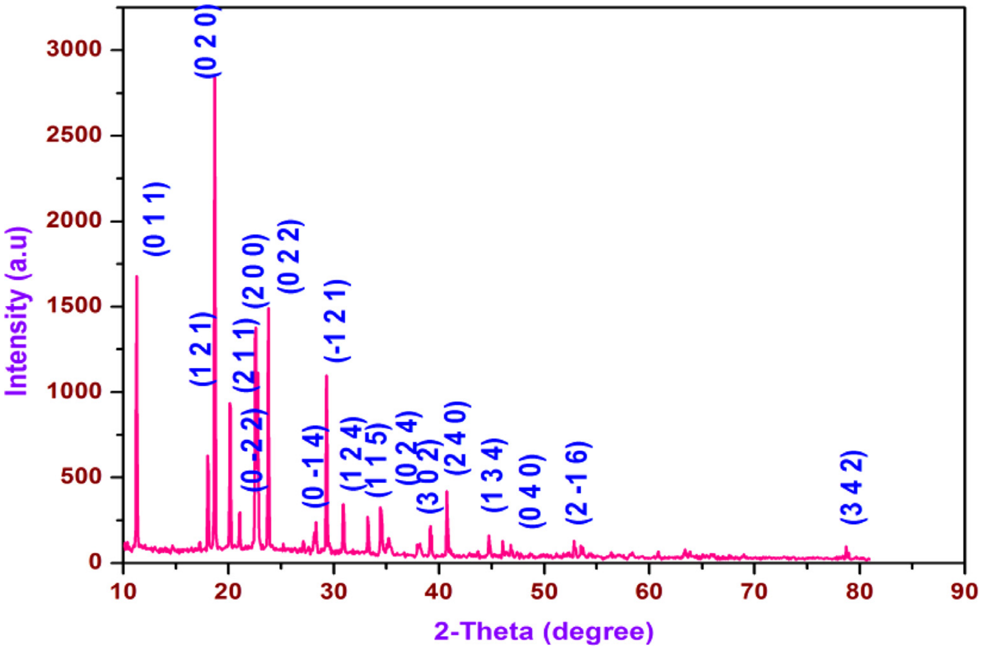

Fig. 3. Powder XRD pattern of 2APCS crystal.

$\alpha = 89.61^{\circ}$; $\beta = 77.31^{\circ}$; $\gamma = 66.56^{\circ}$; and volume of the unit cell is found to be $V = 1321$ Å^3. Hence the grown crystal belongs to Triclinic crystal system with noncentrosymmetric space group P1. The cell parameters of 2-aminopyridine copper sulphate (2APCS) crystals was compared with 2-aminopyridine (2AP), 2-aminopyridine potassium chloride (2APKCL), 2-aminopyridine thiourea zinc sulphate (2APTZS), 2-aminopyridine potassium dihydrogen phosphate (2APKDP), 2-aminopyridine barium chloride (2APBC) are given in Table 1.

FT-IR spectrum of 2APCS was carried out using PERKIN ELMER SPECTRUM RX1 FT-IR spectrometer, by KBr pellet technique between 4000 and 450 cm^{-1}. The observed spectrum was shown in Fig. 4 and their corresponding wave assignments are tabulated in Table 2.

2-Aminopyridine copper sulphate crystal sample was subjected to linear optical study using Lambda-35 UV–visible spectrophotometer. Absorption and transmittance spectrum of 2APCS were cited and shown in Fig. 5. From the figure, no absorption in the whole visible region and 97% transmittance among wavelength 237 nm to 1200 nm have been reported. The lower cut-off occurs at 237 nm for n-π^* transition of NH_2 group. Optical property makes the material vastly utilizable for frequency conversion applications [27].

The florescence spectra of 2APCS crystal were recorded using Cary Eclipse spectrophotometer. Excitation of sample at 260 nm and 570 nm were observed in the emission spectrum indicates that 2APCS crystal contain green luminescence as shown in Fig. 6. The band energy was intended by relation, $E_g = hc/\lambda_e$. Here h and c are constant, λ is the wavelength of florescence. Optical band gap energy $E_g = 3.487$ eV for the 2APCS crystal was calculated.

Mechanical behavior of 2APCS crystal was carried out using SHIMADZU HMV microhardness tester fitted with a diamond pyramidal indenter. The indentation time was fixed at 10 s for various applied loads such as 10, 50 and 100 gs and the

Table 1
Comparison of 2-Aminopyridine derivatives.

Sample	2AP [14]	2APKCL [17]	2APTZS [18]	2APKDP [32]	2APBC [33]	2APCS*
Lattice parameter value (Å)	$a = 11.70 b = 5.67 c = 7.59$	$a = 6.19 b = 6.19 c = 6.19$	$a = 6.45 b = 9.57 c = 13.25$	$a = 7.53 b = 7.53 c = 7.02$	$a = 5.28 b = 5.41 c = 14.89$	$a = 8.84 b = 10.39$ c = 16.14
Volume ($Å^3$)	503	237	818	398	425	1321
Crystal Structure	Monoclinic	Cubic	Orthorhombic	Tetragonal	Orthorhombic	Triclinic
Space Group	$P2_1$	F	$P2_1 2_1\ 2_1$	P1	P1	P1

* Present Work.

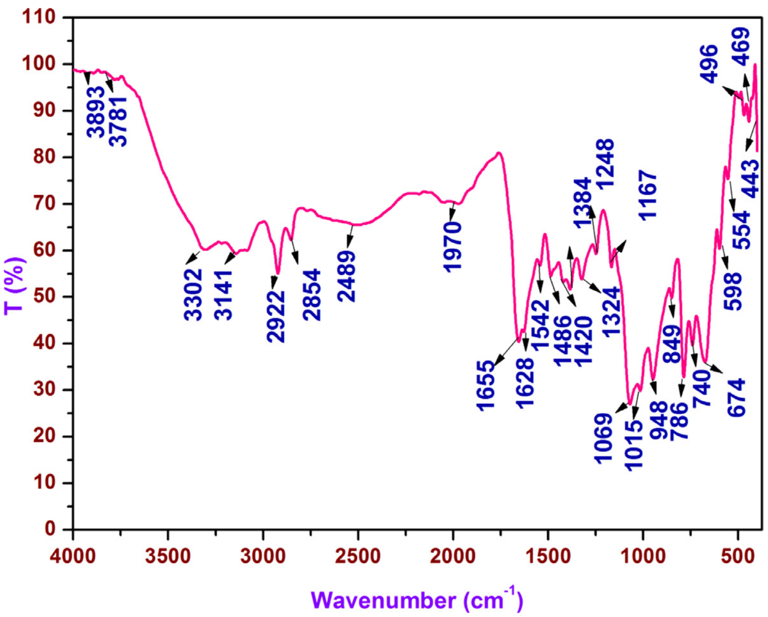

Fig. 4. FT-IR spectrum of 2APCS crystal.

Table 2
Band Assignments of 2APSN crystal.

Wave number cm^{-1}	Assignments
3302,2922, 3141	C-H stretching
2854	N–H stretching
2489	N-H Asymmetric stretching
1655	$C = O$ Asymmetric stretching
1628	O–NO_2 Stretching
1542, 1486	N-C-N Aliphatic stretching
1420	$C = S$ Asymmetric stretching
1384, 849	NO_2 stretching
1324	NH_2 Bending
1167,1015, 948	C–H Out of plane bending
1069	C-N Stretching
786	C–N–C in plane bending
674,740	S-O Stretching
598	$SO_4{}^{2+}$ Asymmetric Stretching
554	C-S-N Symmetric stretching
496, 469	Presence of metal ion

hardness number was calculated using the relation.

$$H_{\upsilon} = 1.8554 \ \frac{P}{d^2} \ \ \mathrm{Kg\,mm^{-2}}$$

Hence P is the load applied in kg and d is the diagonal length of the indented impressions in mm. A plot between the load P and hardness number H_{υ} for 2APCS crystal is shown in Fig. 7. The work-hardening coefficient value was plotted between log p and log d for 2APCS crystals which is shown in Fig. 8. The work hardening coefficient value for 2APCS crystal is 4.77, (which >1.6) comes under the category of soft material and the hardness number decreases with the load is also observed [28].

Third harmonic generation (THG) of 2APCS sample was analyzed using Z-scan technique [29].

Z-scan (Fig. 9) of the on-axis shift by the subsequent equation,

Fig. 10.

$$\Delta T_{p-v} = 0.406 \ (1 - S)^{0.25} \Delta\varphi_0 \tag{1}$$

Where S is the aperture linear transmittance and its relation is given as,

$$s = 1 - exp\left(\frac{-2r_0^2}{\omega_0^2}\right) \tag{2}$$

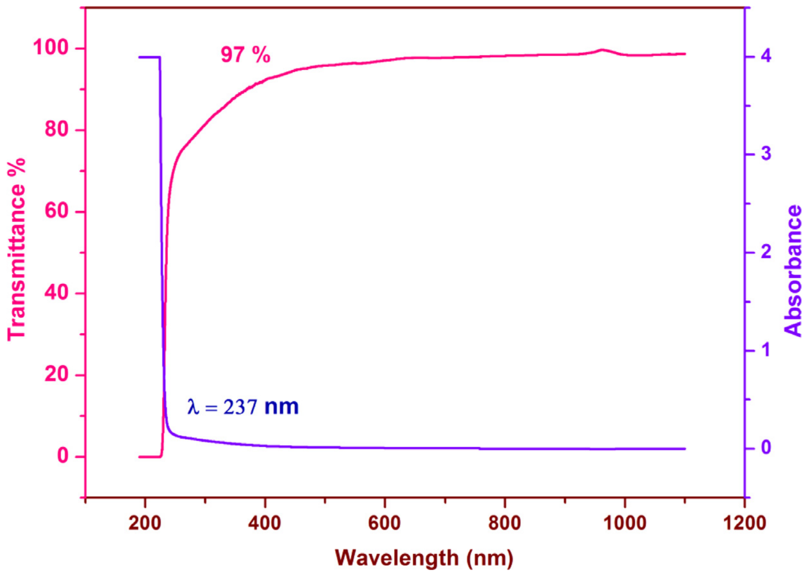

Fig. 5. UV-visible spectrum of 2APCS crystal.

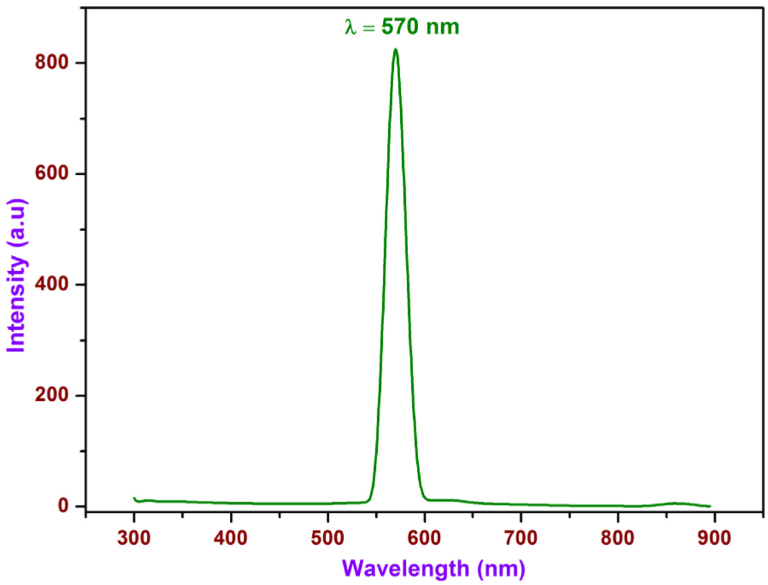

Fig. 6. Emission spectra of 2APCS crystal.

With r_0 indicates the aperture radius and ω_0 indicates the beam radius at the aperture. The nonlinear refractive index (n_2) of the 2APCS crystal is calculated using the formula [30].

$$n_2 = \frac{\Delta\varphi_0\lambda}{2\pi I_0 L_{eff}} \tag{3}$$

Where λ is the laser wavelength, I_0 the intensity of the laser beam at the focus $z = 0$ and the effective sample length $L_{eff} = [1 - exp(\frac{-\alpha L}{\alpha})]$ is the effective thickness of the sample, α is the linear absorption and L is the thickness of the sample. From the open aperture Z-scan data, the non linear absorption coefficient (β) is estimated as

$$\beta = \frac{2\sqrt{2\Delta T}}{I_0 L_{eff}} \tag{4}$$

Where ΔT is the open peak value at the open aperture Z-scan curve. The nonlinear refractive index (n_2) and nonlinear absorption coefficient (β) were estimated. The (β) value will be negative for suitable absorption and positive for two photon absorption. The real and imaginary parts of the third order nonlinear optical susceptibility (χ^3) were determined by the

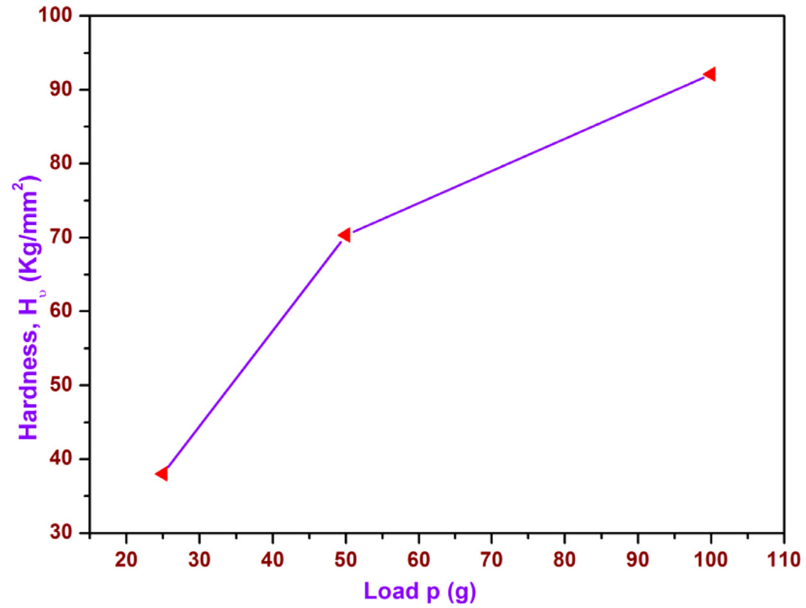

Fig. 7. Variation of Vickers hardness number with applied load of 2APCS crystal.

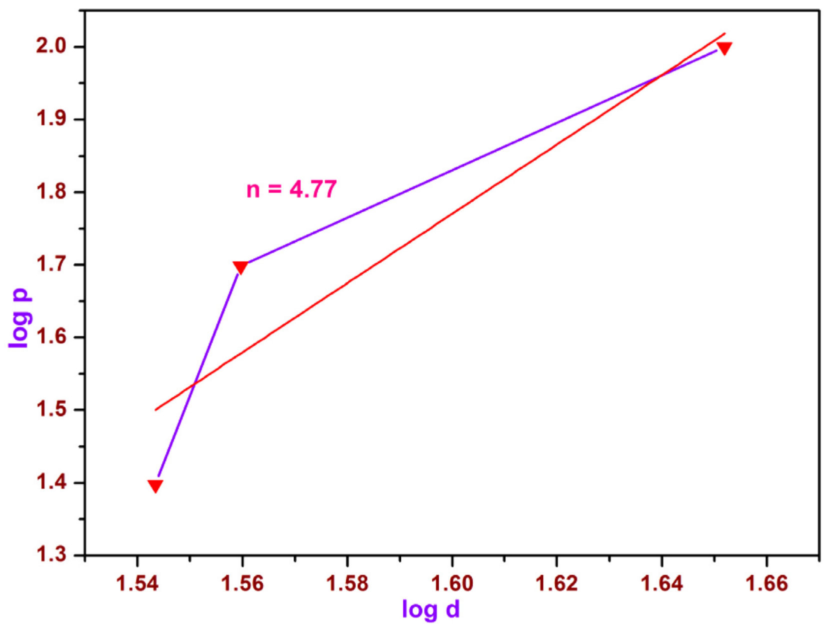

Fig. 8. Plot between logP and log d for 2APCS single crystal.

following relations,

$$Re\left(\chi^3\right)(\mathrm{esu}) = \frac{10^{-4}\left(\varepsilon_0 \mathrm{C}^2 \mathrm{n}_0^2 \mathrm{n}_2\right)}{\pi}\left(\frac{\mathrm{cm}^2}{\mathrm{w}}\right) Im\left(\chi^3\right)(\mathrm{esu}) = \frac{10^{-2}\left(\varepsilon_0 \mathrm{C}^2 \mathrm{n}_0^2 \lambda \beta\right)}{4\pi^2}\left(\frac{\mathrm{cm}}{\mathrm{w}}\right) \tag{6}$$

Where ε_0 is the vacuum permittivity, $\mathbf{n_0}$ is the linear refractive index of the sample and C is the velocity of light in vacuum. The absolute value of (χ^3) was obtained by the relation,

$$\left|(\chi^3)\right| = \left[\left(Re\left(\chi^3\right)\right)^2 + \left(Im\left(\chi^3\right)\right)^2\right]^{\frac{1}{2}} \tag{7}$$

The optical susceptibility of 2APCS is 2.6544×10^{-6} esu. The Corresponding parameter values of laser is listed inTable 3. The susceptibility of 2APCS crystals was tabulated inTable 4. Hence, the 2APCS can be used for various applications in NLO.

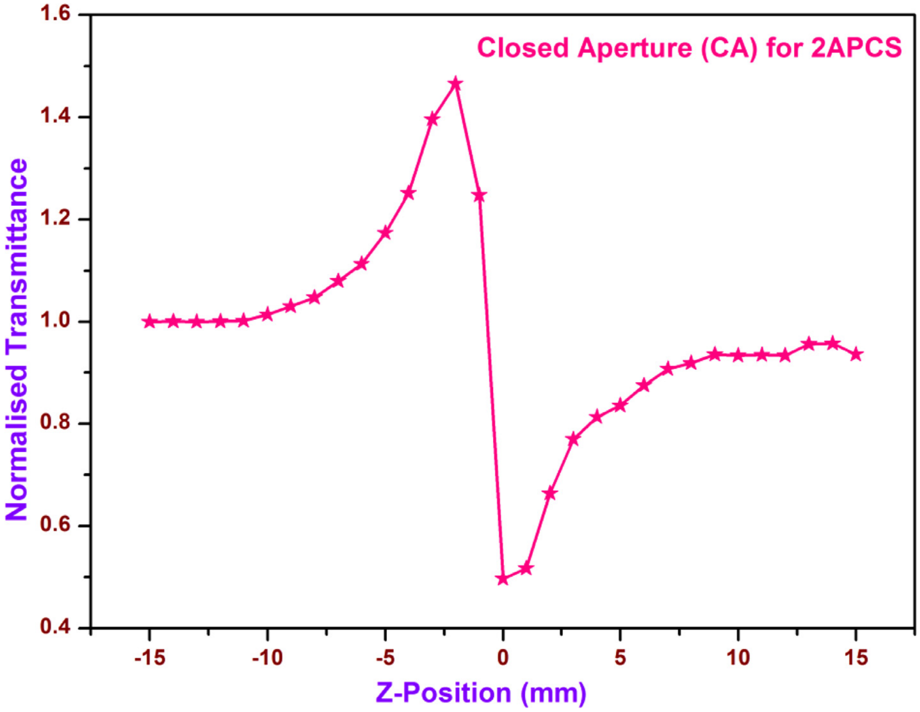

Fig. 9. Closed aperture Z scan plot of 2APCS crystal.

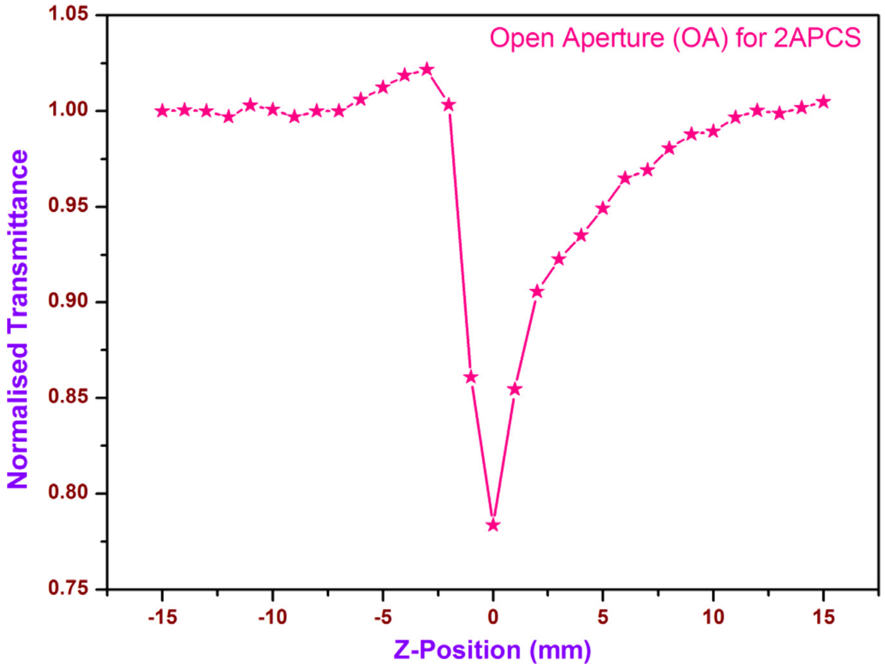

Fig. 10. Open aperture Z scan plot of 2APCS crystal.

The evaluations of antibacterial activity (formation of inhibition zone) of the compound 2-aminopyridine copper sulphate are given in Table 5 and Fig. 11. The zone of inhibition (ZOI) was expressed in mm. The ZOI of the above compound were measured by agar-well-diffusion assay [31] using Enterococcus faecalis, Escherichia coli and Staphylococcus aureus; and compared with the control (Streptomycin). The results revealed that the compound were potentially effective in suppressing microbial growth. among the three tested bacteria, the compound was the most effective retarding microbial growth of

Table 3
Measurement details and the results of the Z- scan technique of 2APCS.

Laser beam wavelength	532 nm and 100 mW
Semiconductor	Continuous wave laser
Path length	675 mm
Beam radius of the aperture (ω_a)	4 mm
Radius of aperture (r_a)	1.25 mm
Beam radius falling on the lens	6 mm
Thickness of sample (L)	1 mm
Laser Power Io	3.47 kW/cm^2
Focal length of lens	103 mm

Table 4
Third order nonlinear optical properties of 2AP complex crystals.

Results Sample Code	n_o	n_2 (cm^2/W)	β (cm/W)	Re$\chi^{(3)}$ (cm^2/W)	Im$\chi^{(3)}$ (cm/W)	X$^{(3)}$ (esu)
2APCS*	1.02624	6.66E-10	2.61E-05	2.64E-06	2.65E-07	2.65449E-06
2-aminopyridine barium chloride (2APBC) [33]	1.0278	1.21E-09	4.39E-05	5.85E-06	4.56E-07	5.87E-06

* Present work.

Table 5
Antimicrobial activity of 2APCS with different concentration.

Name of the organisms	ZOI(mm) Test Extract				ZOI (mm) Standard#
Concentration	25 mg/ Well	50 mg/ Well	75 mg/ Well	100 mg/ Well	30 mg/well
Escherichia coli	9	12	18	20	22
Enterococcus faecalis	10	12	14	16	24
Staphylococcus aureus	11	14	19	22	23

ZOI: Zone of inhibition.
Streptomycin30µg/well.

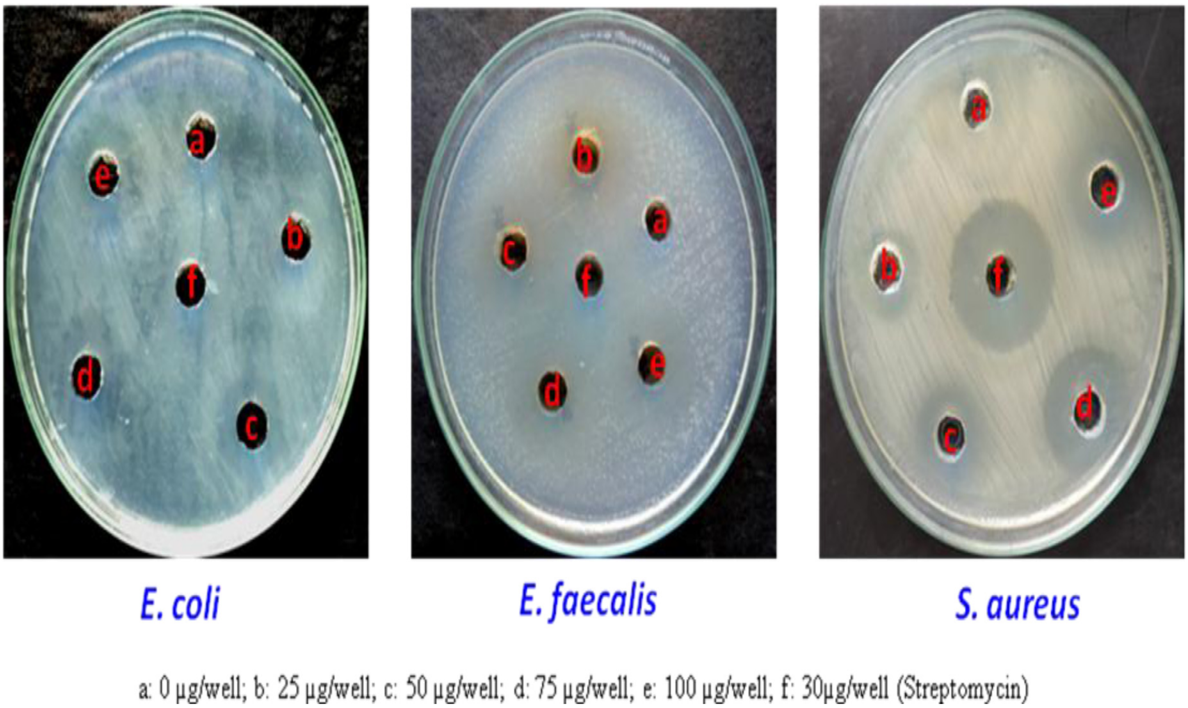

Fig. 11. Antimicrobial Activity of 2APCS.

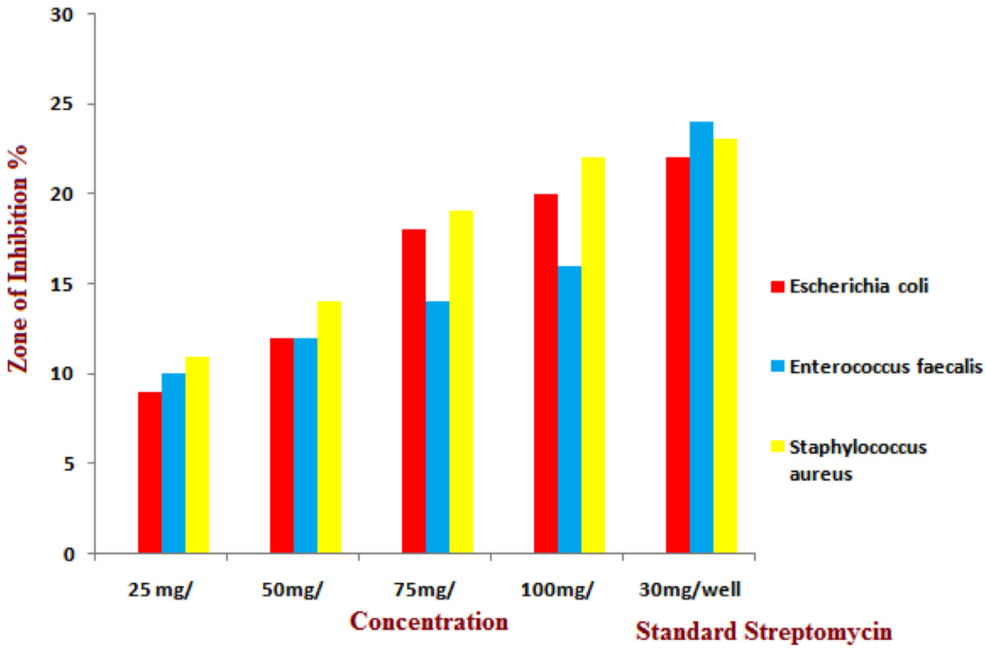

Fig. 12. Bar diagram of antibacterial activity.

the pathogenic bacteria Staphylococcusaureus followed by the other bacteria Enterococcus faecalis and Escherichia coli. But compared to the standard streptomycin our compound was not much effective (Fig. 12).

4. Validation

2APCS crystal was harvested using slow evaporation process in ambient temperature. A well defined sharp Bragg's peaks from powder XRD pattern confirm the crystallinity of 2APCS crystal. Single XRD study reveals 2APCS crystallizes in triclinic system. Various band assignments of 2APCS were identified by FT-IR spectrometer. In linear optical study UV cut off wavelength 237 nm and 97% transparency in the entire visible regions are recorded. In photoluminescence, emission of green laser attests the optical application of the 2APCS crystal. The mechanical coefficient value $n = 4.77$ calculated using Vickers microhardness test, confirms the 2APCS fit in to soft category. THG parameters were calculated for 2APCS using Z- scan technique. In antimicrobial activity, three pathogenic bacteria Enterococcus faecalis (MTCC 439), Escherichia coli (MTCC 443) and Staphylococcus aureus (MTCC 96) the compound 2APCS was most effective retarding microbial growth of the bacteria Enterococcus faecalis (MTCC 439) followed by other bacteria Escherichia coli (MTCC 443) and Staphylococcusaureus (MTCC 96), But compared to the standard streptomycin our compound was not much effective.

Declaration of Competing Interest

None.

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cdc.2019.100319.

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