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Archives of Applied Science Research, 2017, 9 (1):23-30 (http://scholarsresearchlibrary.com/archive.html)



Single Diffusion Gel Growth and Characterization of 4-Acetamidophenol Doped With Tartaric Acid Single Crystal

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ABSTRACT

Single crystals of pure 4-acetamidophenol (AMP) and 4-acetamidophenol (AMP) doped with tartaric acid were grown by single diffusion gel technique. These crystals are characterized by single crystal XRD, powder XRD, FT-IR, FT-Raman, UV-Visible spectroscopy, and SEM with EDX techniques to confirm the presence of tartaric acid in the 4-AMP crystal lattice. The single crystal and powder XRD studies reveal that the doping of tartaric acid disturbs the lattice of parent crystal. This lattice distortion affects the molecular composition of pure 4-AMP crystal. The FT-IR and FT-Raman spectral analysis confirms the presence of various functional groups present in the pure and doped 4-AMP crystals. The optical band gap and the transparency of the grown crystals are found by using the UV-Visible spectroscopy study. The surface morphology of pure and doped crystals have been examined by SEM analysis. The elemental analysis was done by EDX.

Keywords: 4-acetamidophenol, Tartaric acid, Single diffusion gel method, XRD, FT-IR, FT-Raman, UV-Visible spectroscopy, SEM, EDX

INTRODUCTION

The drug compound of paracetamol is also known as their chemical name of acetaminophen or N-acetyl-aminophenol for its antipyretic and analgesic properties. It can be prescribed for fever, headache and mild to medium pain as a single medication or combined with other pharmaceuticals [1,2]. Paracetamol is a derivative of aniline compounds and the para position of -OH group distinguishes the molecules from other acetanilide derivatives [3,4]. Tartaric acid and its derivatives are used widely in pharmaceutical manufacturing to make effervescent salts, and when combined with citrate can improve the taste of oral medications [5]. In the present investigation an attempt was made to grow single crystals of 4-acetamidophenol by doping with tartaric acid of modified composition. This composition modification of material by doping foreign molecule provides very useful information. It is observed that the doping of tartaric acid into pure 4-acetamidophenol affects different characteristics of the parent material. This argument is analyzed by characterized the title compound by single crystal XRD, powder XRD, FT-IR, FT-Raman, UV-Visible spectroscopy, and SEM with EDX analyzes.

MATERIALS AND METHODS

Materials

The materials used for this crystallization are 4-acetamidophenol (98% Alfa Aesar) was purchased from Johnson Matthey company and sodium silicate, ethanol, acetic acid (99. 5%) and tartaric acid were purchased from the Merck, India Ltd, Mumbai.

Single diffusion gel method

Single diffusion gel technique was employed for the growth of pure 4-acetamidophenol and 4-acetamidophenol doped with tartaric acid crystals at room temperature. At first, sodium meta silicate powder is dissolved in water. An acid component added to the solution maintains the pH level of the system. In the present work, acetic and tartaric acids are used to acidify the gel. After the gelation, the other component solution is taken over the set gel. This solution on diffusion through gel medium reacts with the inner reactant and yields the crystals. Sodium meta silicate is used in the present study to forms the gel under a more limited range of conditions. The optimum conditions used in the crystal growth of this method are given in Table 1. The photographic view of the grown crystals is shown in Figure 1.

Parameters	Optimum Conditions				
	Pure 4-AMP	4-AMP doped with tartaric acid			
Density of Na ₂ SiO ₃	1.06 g/cm ³	1.04 g/cm ³			
Concentration of acetic acid	1 M	-			
Concentration of tartaric acid	-	1 M			
Concentration of 4-acetamidophenol	1 M	1 M			
pH of the gel	4.8	4.6			
Gel setting period	2 days	24 hours			
Gel aging	24 hours	24 hours			
Period of growth	3-5 weeks	3-5 weeks			
Temperature	Room temperature	Room temperature			

Table 1: The optimum conditions for grown crystals of pure 4-AMP and 4-AMP doped with tartaric acid.



Figure 1: Photographic view of pure 4-AMP and 4-AMP doped with tartaric acid crystals.

RESULTS AND DISCUSSION

Single crystal XRD analyzes

The molecular composition of material by doping foreign molecule can be analyzed by various techniques. The single crystal X-ray diffraction was done using the SMARTAPEX CCD area-detector diffractometer with Mo K α radiation (λ =0.71073 Å). It gives the unit cell parameter values of the pure 4-AMP and 4-AMP doped with tartaric acid crystals. The unit cell dimensions for doped 4-AMP with tartaric acid crystal is compared with that of pure 4-AMP crystal and are given in Table 2. The molecular structures of 4-acetamidophenol and tartaric acid are depicted in Figures 2a and 2b respectively. It was observed from the single crystal XRD measurement that the pure 4-AMP crystal belongs to monoclinic system while 4-AMP doped with tartaric acid crystal crystallizes in the orthorhombic system. The structural change from monoclinic to orthorhombic by the doping of tartaric acid confirms the presence of tartaric acid in the crystal lattice of 4-AMP crystal.

Cell parameters	Pure 4-AMP	4-AMP doped with tartaric acid
a	7.08 (7) A°	7.14(10) A°
b	9.38(9) A°	9.68(3) A°
с	11.69(5) A°	11.60(3) A°
α	90°	90°
β	97.42°	90°
γ	90°	90°
v	780(6)Å ³	666(4) Å ³
Crystal system	Monoclinic	Orthorhombic

 Table 2: Unit cell parameter values of pure 4-AMP and 4-AMP doped with tartaric acid crystals.

4-acetamidophenol Tartaric acid



Figure 2: Molecular structures of a) pure 4-AMP and b) tartaric acid

Powder XRD analyzes

The X-ray powder diffraction patterns were collected for pure 4-AMP and 4-AMP doped with tartaric acid crystals using a XPERT-PRO X-ray diffractometer with Cu K α (λ =1.54060 Å) radiation. It is used to identify crystalline phases and to qualitatively examine changes in crystallinity. The PXRD diffractograms of pure 4-AMP, pure tartaric acid and the 4-AMP doped with tartaric acid crystals are compared in Figure 3.



Figure 3: Powder X-ray diffraction patterns for pure 4-AMP and 4-AMP doped with tartaric acid crystals.

As seen in the Figure 3, some additional peaks are present in the XRD pattern of doped 4-AMP crystal, showing the presence of additional phases due to doping of tartaric acid. Both single and powder crystal XRD studies show that the dopant has entered into the lattice sites of 4-AMP. Also, the intensity of the 4-AMP doped with tartaric acid crystal increased when compared to pure 4-AMP. The change in intensity suggests that the structure of doped crystal was distorted when compared to pure 4-AMP crystal.

FT-IR and FT-Raman spectroscopy analyzes

The FT-IR spectra of the samples were recorded by using SHIMADZU FT-IR spectrometer in the range 4000-400 cm⁻¹. The sample for this measurement was finally ground and mixed with KBr. The mixture was pressed under vacuum at very high pressure to obtain a transparent disc, which yield good spectra. The FT-Raman spectra for both compounds were recorded by using the BRUKER: RFS 27 Raman spectrometer in the range 4000-400 cm⁻¹. The experimental FT-IR and FT-Raman spectra of pure and doped 4-AMP crystals are shown in Figure 4 and Figure 5 respectively. The detailed wavenumber assignments in both spectra for pure 4-AMP and 4-AMP doped with tartaric acid crystals are given in Table 3.



Figure 4: FT-IR spectra of pure 4-AMP and 4-AMP doped with tartaric acid crystals.



Figure 5: FT-Raman spectra of pure 4-AMP and 4-AMP doped with tartaric acid crystals.

Pure	4-AMP	4-AMP doped with tartaric acid			
FT - IR	FT- Raman	FT - IR	FT - Raman	Assignment	
(ī\u00fc / cm ⁻¹)	(ī\u00fc / cm ⁻¹)	(ī\u00fc / cm ⁻¹)	(ī\u00fc / cm ⁻¹)		
-	-	3539 (s, br)	-	v O-HO	
3325 (s)	3320 (w)	3318 (br)	-	ν N-H	
3163 (br)	3156 (w)	3161 (br)	-	ν О-Н	
3050 (w)	3062 (m)	-	-	υΧ Η	
-	3006 (w)	-	-	υΧ Η	
2981(m)	2980 (m)	-	-	v _{as} -CH ₃	
2931 (m)	-	-	-	v _{as} -CH ₃	
-	-	2941(m)	2947(s)	v C-OH	
2880 (m)	-	2875(w)	-	v _s -CH ₃	
-	-	1701(s, br)	1698 (s)	v C=O(acid)	
1655 (s)	1650 (w)	1645(w)	1640(w)	v C=O	
1613 (m)	1613(w)	-	-	v C=C	
1564 (s)	1562(w)	1561(s)	-	ν C=C + δ N-H	
1508 (s)	1512(w)	-	-	v C=C	
1439 (s)	1439(w)	1441(m)	1440(s)	ν C=C+ β O-H+ β -CH ₃	
-	-	1400(m)	1400(s)	β C-OH	
1370 (m)	1371(w)	1364(m)	-	ν C-C+β C-H	
1325 (w)	1325(w)	-	-	ν C-C+ ν C-N+β C-H	
-	-	1304(m)	-	ν C-N+β C-H+ ν C-O	
1254 (m)	1256(w)	1264(m)	1258(w)	ν C-C+ ν C-N+β C-H	
1227 (m)	1236(w)	-	-	ν C-C+β C-H	
-	-	1207 (m)	1215 (m)	β C-H+ ν C-O	
1171 (w)	1169(w)	-	-	ν C-C+ρ-CH ₃ +β C-H	
-	-	1138(s)	1142(s)	ρ-CH ₃ +β C-H	
1107 (w)	1104(w)	-	-	ν C-C+β C-H	
-	-	1069(w)	1066(w)	β С-Н	
1015 (w)	1014(w)	-	-	ν C-C+β C-H	
966 (s)	968(w)	995(m)	994(m)	γ O-H+β C-H	
950 (sh)	950(sh)	950(m)	950(w)	Ring breathing	
-	-	907 (m)	907(w)	β С-ОН	
837 (s)	832(w)	845(m)	838(w)	ү С-Н	
804 (m)	797(w)	785(m)	783(w)	γ C-H	
718 (w)	712(w)	-	-	γ C-H+ ω N-H	
685 (s)	685(w)	677(m)	677(w)	γ C-H+ ω N-H	

Table 3: Observed wavenumbers in both spectra and their assignments for pure 4-AMP and 4-AMP doped with tartaric acid crystals.

Vibrations of 4-acetamidophenol

The 4-acetamidophenol molecule containing the three fundamental functional groups that is, benzene ring, amide (HN-CO-CH₃) and hydroxyl (O-H) groups (Figure 2a).

Benzene ring vibrations

C-H group vibrations: In the benzene ring, all the CH stretching vibrations have the infrared peaks in the region 3100-3000 cm⁻¹ [6]. Accordingly, the symmetric stretching vibration of the C-H group is assigned at 3050 cm⁻¹ and 3062, 3006 in FT-IR and FT-Raman spectra of pure 4-AMP crystal. As expected, the bands due to C-H stretching vibrations of 4-AMP doped with tartaric acid crystal are not observed in the IR and Raman spectra due to the fact that the respective bands merge with broad band centered around 3000 cm⁻¹ due to hydrogen bonding. The C-H in plane bending vibrations usually occurs in the region 1430-990 cm⁻¹ [6]. In this study, the FT-IR peaks at 1370, 1325, 1254, 1227, 1171, 1107 and 1015 cm⁻¹ are assigned to C-H in-plane-bending mode of pure 4-AMP crystal. Also, the same mode is attributed to the pure 4-AMP crystal at 1371, 1325, 1256, 1236, 1169, 1104, 1014 cm⁻¹ in FT-Raman spectrum. The wavenumbers at 1400, 1364, 1304, 1264, 1207, 1138, 1069, 995 cm⁻¹ and the wavenumbers at 1258, 1215, 1142, 1066 cm⁻¹ in IR and Raman spectra respectively are identified as the β (C-H) mode of 4-AMP doped with tartaric acid crystal. The aromatic out-of-plane C-H bending absorption bands occur between 900-690 cm⁻¹ regions [7]. In the present study, the peaks are identified at 837, 804, 718 cm⁻¹ and 832, 797, 712 cm⁻¹ in both spectra confirm

the presence of C-H out of plane bending vibrations of pure 4-AMP crystal. For the doped 4-AMP crystal γ (C-H) mode is attributed at 845, 785 cm⁻¹ and 838, 783 cm⁻¹ in FT-IR and FT-Raman spectra respectively.

C=C and C-C group vibrations: The C=C ring stretching modes are observed between the region 1650-1430 cm⁻¹ [8]. This C=C aromatic stretching vibrations are noted for the pure 4-AMP crystal at 1613, 1564, 1508, 1439 cm⁻¹ in FT-IR spectrum. This mode is closely observed in FT-Raman spectrum of pure 4-AMP crystal. The wavenumbers at 1441 cm⁻¹ in both spectra is assigned for the C=C ring stretching modes of doped 4-AMP crystal.

The expected range of C-C vibration lies between 1400-1300 cm⁻¹ [8]. In the present work, it is assigned at 1370 and 1325 cm⁻¹ in FT-IR spectrum and at 1371, 1325 cm⁻¹ in FT-Raman spectrum of pure 4-AMP crystal. The C-C stretching mode for doped crystal is observed at 1364 cm⁻¹ in FT-IR spectrum only and there is no counterpart in FT-Raman spectrum for this mode. The ring breathing mode is identified at 950 cm⁻¹ in the both spectra of the pure and doped 4-AMP crystals respectively.

Amide group vibrations

The N-H stretching of amide group is observed at 3300-3250 cm⁻¹ and its bending appears at 1560-1530 cm⁻¹. Also, N-H wagging band appears at 750-650 cm⁻¹[9]. In the present work, the N-H stretching and bending modes of pure 4-acetamidophenol are identified as the sharp intense peaks at 3325 cm⁻¹ (IR), 3320 cm⁻¹ (Raman) and 1564 cm⁻¹ (IR),1562 cm⁻¹ (Raman) respectively. At the same time, the stretching and bending modes of N-H group for doped 4-AMP crystal is appeared in FT-IR spectrum at 3318 cm⁻¹ and 1561 cm⁻¹ respectively. The N-H wagging mode of pure 4-AMP crystal is observed at 718, 685 cm⁻¹ in the both spectra. This wavenumbers are shifted to 785, 677 cm⁻¹ in both spectra of doped 4-AMP crystal.

C-N group vibration: Also the C-N stretching vibration of secondary aromatic amide is absorb strongly at 1342-1320 cm⁻¹ and 1315-1250 cm⁻¹ in the IR spectrum [9]. The pure 4-AMP crystal absorbs the bands at 1325 cm⁻¹ and 1254 cm⁻¹ in the both spectra. This mode is shifted to those in the doped crystal at 1304 cm⁻¹ and 1264 cm⁻¹ for C-N stretching mode of doped 4-AMP crystal.

Methyl group vibrations

In methyl group (-CH₃), the C-H stretching mode has lower wavenumber than those of aromatic ring (3100-3000 cm⁻¹). Moreover, the antisymmetric stretch is usually observed at higher wavenumber side than the symmetric stretch. In this present work, the -CH₃ symmetric stretching frequency is assigned at 2880 cm⁻¹, whereas the antisymmetric stretching mode are assigned at 2931 cm⁻¹ in the FT-IR spectrum of 4-acetamidophenol. The doped 4-AMP crystal has the symmetric and antisymmetric stretching mode of -CH₃ group at 2875 cm⁻¹ (IR) and 2981 cm⁻¹ (IR), 2980 cm⁻¹ (Raman).

The deformation of $-CH_3$ group is usually observed in the range 1450-1400 cm⁻¹ for methyl substituted aromatic rings [10]. Accordingly, the peaks at 1439 cm⁻¹ in both spectra is assigned to $-CH_3$ in-plane bending deformation vibrations of pure and doped 4-AMP crystals. The peaks at 1171 cm⁻¹ (FT-IR) are assigned to $-CH_3$ rocking modes for pure 4-AMP crystal and it is shifted to 1138 cm⁻¹(FT-IR), 1142 cm⁻¹(FT-Raman) for doped 4-AMP crystal.

C=O group vibration: The C=O stretching vibration is assigned in the literature as a very strong band at 1661 cm⁻¹ in FT-IR spectrum [11]. The C=O stretching mode of pure 4-AMP is found at 1655 cm⁻¹ and 1650 cm⁻¹ in FT-IR and FT-Raman spectra respectively. For 4-AMP doped with tartaric acid crystal is attributed at 1645 cm⁻¹ and 1640 cm⁻¹ in both spectra respectively.

Hydroxyl group vibrations

The free -OH group of phenol absorbs a band at 3615 cm⁻¹ while the associated -OH group has a stretching wavenumber in the range 3250-3200 cm⁻¹[12,13]. In the present work, the broad bands occur at 3163 cm⁻¹, 3161 cm⁻¹ are attributed to the O-H stretching mode of the pure and doped 4-AMP crystals respectively. The O-H in- plane and out- of -plane bending wavenumbers normally occurs in the region between 1440-1395 cm⁻¹ and 960-875 cm⁻¹ respectively [14]. In the present study, bands at 1439 cm⁻¹ and 966 cm⁻¹ in the FT-IR spectrum is assigned to the O-H in plane and out of plane bending wavenumbers of pure 4-AMP crystal respectively. These wavenumbers are upshifted to 1441 cm⁻¹ and 995 cm⁻¹ for doped 4-AMP crystal. The downshifting and upshifting the wavenumber of stretching and bending modes of doped crystal confirms the interaction of tartaric acid with the 4-AMP by the formation of hydrogen bonds. The broadband centered on 3000 cm⁻¹ also supports the presence of hydrogen bonding network in the title crystal.

Vibrations of tartaric acid

The dopant element of tartaric acid has two hydroxyl (OH) and two carboxylic acid (COOH) groups (Figure 2b).

Carboxylic group vibrations

The C=O vibration of -COOH group of L-tartaric acid crystal occurs at 1737 cm⁻¹ [15]. In the present work, this mode is identified at 1701 cm⁻¹ and 1698 cm⁻¹ in FT-IR and FT-Raman spectra of doped 4-AMP crystal respectively. This shift is due to the presence of predominant nature of tartaric acid molecule in the 4-AMP crystal. This mode is not identified in both spectra of pure 4-AMP crystal.

Hydroxyl group vibrations

The O-H stretch from CO-OH vibration is observed at 3100-2800 cm⁻¹[16]. In the present study, the wavenumbers are observed at 2941 cm⁻¹ and 2947 cm⁻¹ in the FT-IR and FT-Raman spectra of doped 4-AMP crystal respectively. The O-H in plane and out of plane bending wavenumbers normally occurs in the region between 1440-1395 cm⁻¹ and 960-875 cm⁻¹ respectively [14, 15]. In the present study, the doped 4-AMP has strong bands at 1400 cm⁻¹ and 907 cm⁻¹ in both spectra which is attributed to O-H in plane and out-of-plane deformation modes respectively.

C-O vibrations: The C-O stretching of carboxylic acids appears near the region 1320-1210 cm⁻¹ in the spectra [16, 17]. In the present study, the C-O stretching of tartaric acid is identified at 1304, 1207 cm⁻¹ in the IR and 1215 cm⁻¹ in the Raman spectra of doped 4-AMP crystal. But this mode is not appearing in the pure 4-AMP crystal. The absence of above three vibrational modes in both spectra of pure 4-AMP crystal is strongly support the presence of tartaric acid in the crystal lattice of parent crystal.

Ultraviolet Visible spectroscopy analyzes

The UV-Visible spectroscopy analyzes is one of the most frequently employed techniques to analyze the pharmaceutical compound. The optical transmittance and absorption spectrum of grown crystals has been recorded with SHIMADZU-UV1800, double beam spectrometer. Transmittance and absorbance data were observed for both crystals in the wavelength range 200-1100 nm insteps of 1 nm. The slit width chosen was 1 nm. The wavelength rate was in medium mode. The observed values of absorbance were recorded and stored in the memory of a computer and plotted. The absorbance spectrum of 4-AMP and 4-AMP doped with tartaric acid crystals is shown in Figure 6. The maximum absorbance wavelength (λ_{max}) for pure and doped 4-AMP crystals are found to be at 248 nm, 218 nm and the lower cut-off wavelength is observed at 302 nm, 245 nm respectively.





From the absorbance spectrum, the pure and doped 4-AMP crystals shows the good transmittance in the entire range of visible region. The band gap of the crystals were estimated by using the following the relation [18].

$$E_g = \frac{1.243 \times 10^3}{\ddot{e}_{max}}$$

The optical band gap was evaluated by plotting $(\alpha hv)^2 vs$. hv as shown in Figure 7 and extrapolating the linear portion of absorption edge $(\alpha hv)^2$ to the photon energy axis gives the optical band gap of the crystal. The band gap value of the crystal was found to be as 4 eV and 5.1 eV for pure and doped 4-AMP crystals respectively. The band gap value

increased while the dopant was added to pure 4-AMP. The high value of band gap indicates that the grown crystals may be act as a typical of dielectric material.



Figure 7: Variation of photon energy (hv) with $(\alpha hv)^2$ of pure 4-AMP and 4-AMP doped with tartaric acid crystals.

SEM with EDX Analysis

Morphologies of pure and doped crystals were investigated by using SEM analysis. The surface morphology and elemental analysis of pure and doped 4-AMP with tartaric acid crystals have been analyzed by CARLZEISS EVO18 scanning electron microscope. The pure 4-AMP exhibited regular shape with smooth surface. The doped 4-AMP crystal also exhibited regular shape but the shape was different with those of the pure 4-AMP crystal. The SEM microphotographs of pure 4-AMP and 4-AMP doped with tartaric acid crystals are shown in Figure 8. The EDX spectrum of 4-AMP doped with tartaric acid crystal is shown in Figure 9. The peaks show the presence of Carbon, Nitrogen and Oxygen in the Crystals. Table 4 shows the elemental and atomic percentage of the elements C, N and O. It was observed that the atomic % of C, N and O are 20.14, 26.25 and 49.69 respectively. The result shows that the atomic % of C and O increases due to the addition of dopant and also reveals the presence of tartaric acid in the crystals.



Figure 8: SEM microphotographs of pure 4-AMP and 4-AMP doped with tartaric acid crystals.



Figure 9: The EDX spectrum of 4-AMP doped with tartaric acid crystal.

Table in Elemental composition of a right about whith an are of a crystall.				
Element	Atomic percentage (%)	Weight percentage (%)		
Carbon	20.14	6		
Oxygen	49.69	18.18		
Nitrogen	26.25	9.08		
Sodium	3.91	1.25		

 Table 4: Elemental composition of 4-AMP doped with tartaric acid crystal.

CONCLUSION

Pharmaceutical single crystals of pure 4-AMP and 4-AMP doped with tartaric acid were grown by using the gel method. The harvested colorless transparent crystals were carried out to the single crystal XRD, powder XRD, FT-IR, FT-Raman, UV-Visible spectroscopy and SEM with EDX analyzes. The single crystal XRD and powder XRD analyzes reveals that doping of tartaric acid affects the crystal structure of the parent crystal. The downshifting and upshifting the wavenumber of stretching and bending modes of doped crystal in FT-IR and FT-Raman spectra confirms the interaction of tartaric acid with the 4-AMP by the formation of hydrogen bonds. The optical band gap of doped 4-AMP crystal is decreased in the presence of tartaric acid. It is also confirm the dopant molecule of tartaric acid enter into the 4-AMP crystal lattice. The surface morphology was also studied by SEM analysis and the results indicate that the doped crystal has different morphology from the pure 4-AMP crystal. The elemental composition of doped 4-AMP crystal was analyzed by EDX spectrum. The result shows that the atomic percentage of C and O increases due to the addition of dopant and also reveals the presence of tartaric acid in the crystals.

ACKNOWLEDGEMENT

The authors sincerely acknowledge their thanks to the Management and Principal of Devanga Arts College, Aruppukottai for their permission and encouragement during their research work.

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