



Vapor Diffusion Growth and Characterization of Aspirin – Perchloric acid Complex Crystal

 C. Muthuselvi¹, S. Pandiarajan¹ and R.V. Krishnakumar²
¹Department of Physics, Devanga Arts College, Aruppukottai, 626 101, Tamilnadu, India.

²Department of Physics, Thiagarajar College, Madurai, 625 009, Tamilnadu, India.

ARTICLE INFO

Article history:

Received: 15 April 2016;

Received in revised form:

25 May 2016;

Accepted: 31 May 2016;

Keywords

 Vapor diffusion method,
 Aspirin–Perchloric acid
 complex,
 Powder XRD,
 FT–IR, FT–Raman,
 UV–Visible
 spectroscopy,
 SEM.

ABSTRACT

The aspirin–perchloric acid complex crystal is grown by the vapor diffusion method for the first time. The colorless needle shaped crystals are obtained after 5 weeks duration. These crystals are characterized by powder XRD, FT–IR, FT–Raman, UV–Visible spectroscopy, SEM and melting point studies to confirm the formation of complex crystal of aspirin –perchloric acid. The powder XRD, FT–IR and FT–Raman studies reveal the presence of perchloric acid with the medicinally important drug of aspirin in the complex form. The UV–Visible spectroscopy study shows that the optical window is found to be reduced in the presence of perchloric acid. But there is no change in the transparency of the both crystal in the visible region. The SEM analysis reveals the complex crystal has the distinct morphology in shape from the pure aspirin crystal. The melting point of complex crystal is found to be 110°C. Finally, these studies conclude that mixing of perchloric acid with aspirin changes the all physico-chemical properties of the pure drug compound of aspirin. The property change of aspirin–perchloric acid complex can be used to improve the medicinal activity of the drug compound in the biological systems.

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Introduction

The salicylate drug of acetylsalicylic acid is also called as aspirin. It can act as an effective analgesic to reduce the mild headache pain, toothache, muscle pain, and joint pain [1]. Also, it behaves as an antipyretic drug to reduce fever and an anti-inflammatory medication which is capable of reducing swelling and redness associated with inflammation. The low doses of aspirin may be given to the patient immediately after a heart attack to reduce the risk of another heart attack or of the death of cardiac tissue [2–4]. Now a day, the effect of aspirin on colorectal cancer has been widely studied by the many researchers and they conclude that the regular use of aspirin reduces the long-term risk of Colorectal Cancer Risk (CRC) incidence and mortality [5]. Most of the drugs are delivered to patients in crystalline form [6]. The crystals of metal complexes with aspirin have been found to be some additional medical activities such as antiulcer, anticancer, antimutagenic and antioxidative in biological systems [7, 8]. In the present study, we are interested to grow the medicinal important aspirin drug crystal doping with the perchloric acid by vapor diffusion method. The grown crystals are characterized by the powder XRD, FT–IR, FT–Raman, UV–Visible spectroscopy, SEM and melting point studies. These results are compared with the pure aspirin drug crystal and they are summarized in the present study.

Materials and Methods

The raw materials used for crystallization (Aspirin, Perchloric acid and Ethanol) were purchased from the Merck India Ltd, Mumbai.

Crystal growth by vapor diffusion method

The aspirin crystals and aspirin–perchloric acid complex crystals were grown by using the vapor diffusion method. In this method, aspirin and perchloric acid were taken in the 1:1

stoichiometric ratio and they are dissolved in ethanol as a solvent in a small beaker (50 ml). This mixture is placed inside a larger beaker (250 ml) that contains a small volume of a solvent system (water) in which the sample is insoluble. The outer beaker was sealed. Vapor from the solvent of the inner beaker diffuses into the solution in the outer beaker, without disturbing the vessel causing the mixture to grow crystals. Since the rate of evaporation of ethanol slow, good quality crystals were formed after 5 weeks. The arrangement of vapor diffusion method is illustrated in Fig.1. The photographic view of the harvested colorless rectangular and needle shaped, aspirin and aspirin–perchloric acid complex crystals respectively are shown in Fig. 2. The molecular structures of pure aspirin and aspirin-perchloric acid complex crystals are depicted in Fig. 3.

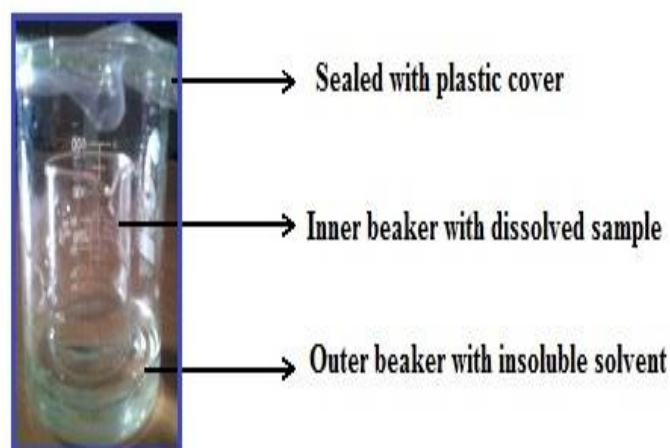


Figure 1: Arrangement of vapor diffusion method.

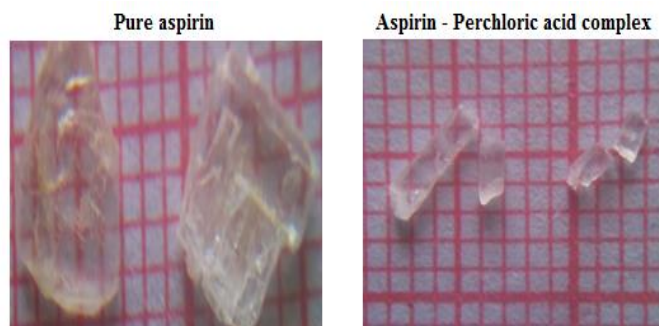


Figure 2: Photographic view of the harvested pure aspirin and aspirin-perchloric acid complex crystals.

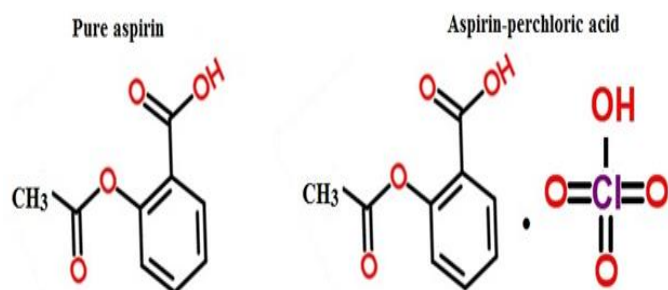


Figure 3: Molecular structures of pure aspirin and aspirin-perchloric acid complex crystals.

Results and Discussions

The aspirin crystals and aspirin-perchloric acid complex crystals were grown by the vapor diffusion method. These crystals are characterized by the powder XRD, FT-IR, FT-Raman, UV-Visible spectroscopy SEM and melting point studies.

Powder X-ray Diffraction Analysis

The powder X-ray diffraction study of aspirin and aspirin-perchloric acid crystalline samples were carried out, using XPERT-PRO X-ray diffractometer with Cu K α ($\lambda = 1.54060 \text{ \AA}$) radiation. The X-ray powder diffraction pattern for the pure aspirin and aspirin-perchloric acid complex crystals are shown in Fig. 4. The sharp and well defined peaks indicate the crystalline nature of the compound. The complex crystalline nature has been identified by comparing the interplaner spacing and 2θ values of the powder pattern with the pure aspirin crystal. The d spacing and 2θ values of complex crystal are different from that of pure aspirin and they are shown in Table 1. This result confirms the formation of aspirin-perchloric acid complex crystal.

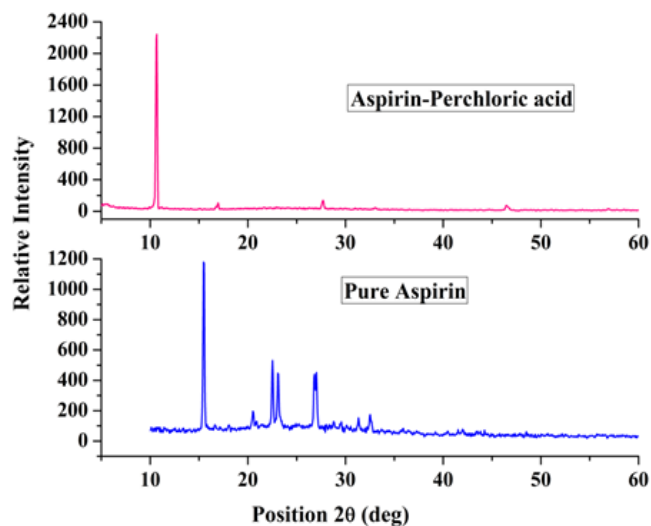


Figure 4: Powder diffraction pattern of pure aspirin and aspirin-perchloric acid complex crystals.

Vibrational Analysis

The infrared spectroscopy and Raman spectroscopy analyzes are employed here for the identification and assignment of the various functional groups present in the title compound. The FT-IR spectrum of the aspirin and aspirin-perchloric acid crystals were recorded using SHIMADZU FT-IR spectrometer in the range $4000-400 \text{ cm}^{-1}$. The sample for this measurement was finely ground and mixed with KBr. The mixture was pressed under vacuum at very high pressure to obtain a transparent disc, which yields good spectra. The FT-IR spectra of pure aspirin and aspirin-perchloric acid complex crystals are shown in Fig.5. The FT-Raman spectra of pure aspirin and aspirin-perchloric acid complex crystals were recorded over the range $4000-80 \text{ cm}^{-1}$ with a resolution of 2 cm^{-1} using the BRUKER RFS 27 FT-Raman spectrometer. The source used in this device was the Nd : YAG laser operated at 1064 nm with the incident power of 100 mW for excitation. The FT-Raman spectra of pure aspirin and aspirin-perchloric acid complex crystals are shown in Fig.6. The title compound has C=O (acid), C=O (ester), C-O (acid), C-O (ester), O-H, -CH $_3$, benzene ring and HClO $_4$ functional groups (Fig.3). The detailed assignments of absorption bands/peaks observed in the FT-IR and FT-Raman spectra of pure aspirin and aspirin-perchloric acid complex crystals are shown in the Table 2. The wavenumber assignment of aspirin and perchloric acid are available on the earlier documented literature [9-11].

Table 1: Powder XRD data of pure aspirin and aspirin-perchloric acid complex crystals.

Pure aspirin				Aspirin-perchloric acid complex			
Position [°2 θ]	d-spacing [Å]	FWHM Left [°2 θ]	Rel. Int. [%]	Position [°2 θ]	d-spacing [Å]	FWHM Left [°2 θ]	Rel. Int. [%]
5.42	16.29	1.50	11.29	5.00	17.57	3.00	3.54
7.37	11.98	0.13	97.96	10.63	8.30	0.15	100.00
10.57	8.36	0.13	67.64	16.93	5.23	0.10	3.79
15.17	5.83	0.19	100.00	27.65	3.22	0.20	5.06
23.05	3.85	0.19	11.65	33.01	2.71	0.2	1.18
26.53	3.35	0.51	5.08	46.50	1.95	0.36	2.25
31.00	2.88	0.30	5.50	56.88	1.61	0.22	0.97

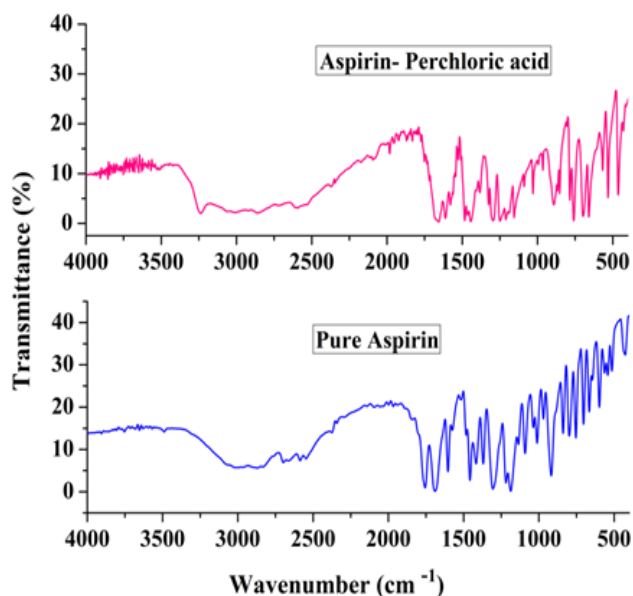


Figure 5: FT-IR spectra of pure aspirin and aspirin-perchloric acid complex crystals.

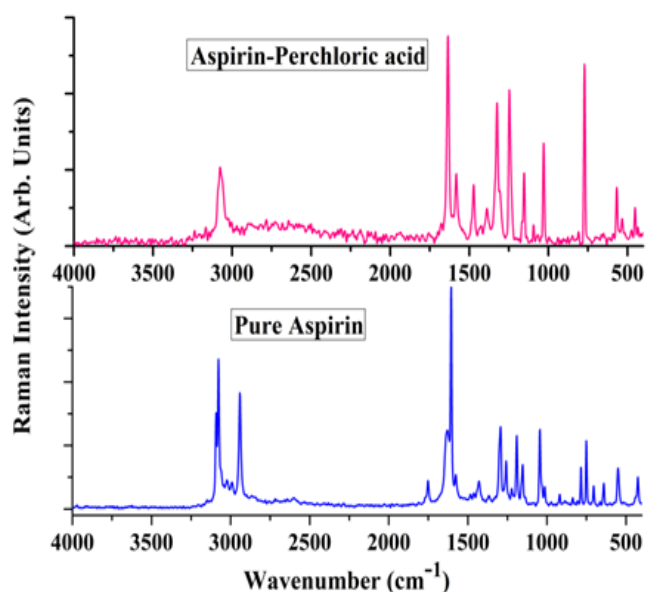


Figure 6: FT-Raman spectra of pure aspirin and aspirin-perchloric acid complex crystals.

C=O Vibrations

The two C=O peaks should be expected for the pure aspirin crystal between the region $1800\text{--}1680\text{ cm}^{-1}$, one occurring at a slightly higher frequency due to ester type C=O vibrations and the other at slightly lower frequency due to the acid type C=O vibration [12]. In the present work, the acid C=O stretching mode of aspirin is observed at $1692, 1694\text{ cm}^{-1}$ in FT-IR and FT-Raman spectra respectively. But this mode is shifted to $1685, 1687\text{ cm}^{-1}$ in the IR and Raman spectra of the aspirin-perchloric acid complex crystal due to the fact that the aspirin linkage with the perchloric acid through the carboxylic group by the formation of O-H...O hydrogen bonds. This is due to the engagement of double bonded O atom of carboxylic group with the O-H group in the perchloric acid. Also the stretching of ester C=O group is identified as a strong band at 1755 cm^{-1} in the IR and as a medium band at 1751 cm^{-1} in the Raman spectra of pure aspirin crystal. In the complex crystal this mode is observed at 1748 cm^{-1} and 1745 cm^{-1} in the both spectra respectively.

Aromatic acids have a rocking vibration of the CO₂ group as a strong band at $570\text{--}545\text{ cm}^{-1}$. Also the deformation mode occurs in the region $620\text{--}610\text{ cm}^{-1}$ for CO₂ group [13]. In the present investigation the medium bands are observed at $563, 551\text{ cm}^{-1}$ the IR and Raman spectra respectively for pure aspirin crystal and 567 cm^{-1} in both spectra of aspirin complex crystals are assigned to CO₂ rocking vibration. The deformation mode of CO₂ group is attributed due to the shoulder band at 615 cm^{-1} in FT-IR spectra of complex crystal and this band disappears in the pure aspirin spectrum.

C-O Vibrations

The C-O stretching of carboxylic acids appears near the region $1320\text{--}1210\text{ cm}^{-1}$ in the spectra [14, 15]. In the present study, the C-O stretching of carboxylic acid is identified due to the bands at $1306, 1219\text{ cm}^{-1}$ in the IR and $1293, 1223\text{ cm}^{-1}$ in the Raman spectra of pure aspirin. But this mode is shifted to $1296, 1209\text{ cm}^{-1}$ in the IR spectrum for the complex molecule due to the involvement of OH group in the in-plane bending vibrations of carboxylic group. This mode is identified as a very weak band at 1206 cm^{-1} in the FT-Raman spectrum of complex crystal.

The C-O stretching has the bands near 1095 cm^{-1} and 1016 cm^{-1} for ester group [15]. In the present study the stretching modes of C-O ester group are identified at $1092, 1013\text{ cm}^{-1}$ and $1088, 1028\text{ cm}^{-1}$ in IR spectra of the pure aspirin and complex crystals respectively. Also, the corresponding Raman bands are identified at 1014 cm^{-1} and $1093, 1030\text{ cm}^{-1}$ for both crystals respectively.

O-H Vibrations

The O-H stretch from CO-OH vibration is observed at $3100\text{--}2800\text{ cm}^{-1}$ [16]. In the present study, these wavenumbers are observed at $3080, 2999, 2870\text{ cm}^{-1}$ in the FT-IR and $3077, 3023, 2992, 2941\text{ cm}^{-1}$ in the FT-Raman spectra for pure aspirin. These wavenumbers of pure aspirin for this mode are compared with the aspirin-perchloric acid complex crystal and are identified at $3061, 3009, 2940, 2859\text{ cm}^{-1}$ and 3075 cm^{-1} in the both spectra respectively. The O-H in plane and out of plane bending wavenumbers normally occurs in the region between $1440\text{--}1395\text{ cm}^{-1}$ and $960\text{--}875\text{ cm}^{-1}$ respectively [14, 15]. In the present study, pure aspirin has strong bands at 1418 cm^{-1} and 918 cm^{-1} in the FT-IR spectrum which is attributed to O-H in plane and out-of-plane deformation modes respectively. These wavenumbers are upshifted to 1443 cm^{-1} and 964 cm^{-1} for aspirin-perchloric acid complex crystal. The downshifting and upshifting the wavenumber of stretching and bending modes of O-H group of complex crystal confirms the interaction of perchloric acid with the aspirin through the -COOH group by the formation of hydrogen bonds. The broad band centered on 3000 cm^{-1} also supports the presence of hydrogen bonding network in the title crystal.

Benzene ring vibrations

The aromatic C-H stretching bands appear in the region $3100\text{--}3000\text{ cm}^{-1}$ [17-20]. In the present study the observed bands at 3080 cm^{-1} and $3092, 3077, 3023\text{ cm}^{-1}$ in the IR and Raman spectra respectively are assigned to C-H stretching of pure aspirin crystal. Also the bands at $3061\text{ cm}^{-1}, 3009\text{ cm}^{-1}$ and $3165, 3075\text{ cm}^{-1}$ in the IR and Raman spectra respectively are attributed to C-H stretching vibrations of the aspirin-perchloric acid complex crystal. The ring breathing mode for ortho substituted benzene ring is normally observed at 1040 cm^{-1} [15]. In the present study this mode is exactly observed in both spectra of pure aspirin and aspirin-perchloric acid complex crystals.

Table 2: Observed wavenumbers and their assignments for pure aspirin and aspirin– perchloric acid complex crystals in the both spectra.

Pure Aspirin		Aspirin–Perchloric acid		Assignment
FT – IR ($\bar{\nu}$ / cm^{-1})	FT – Raman ($\bar{\nu}$ / cm^{-1})	FT – IR ($\bar{\nu}$ / cm^{-1})	FT – Raman ($\bar{\nu}$ / cm^{-1})	
–	–	3237 (vs, br)	3233 (w)	ν O–H \cdots O
–	–	–	3165 (vw)	ν C–H
–	3092(m)	–	–	ν C–H
3080 (s, br)	3077(vvs)	3061 (s, br)	3075 (s)	ν C–H; ν C–OH
–	3023(w)	3009 (s, br)	–	ν C–H; ν C–OH
2999 (s, br)	2992 (w)	–	–	ν_{as} –CH ₃ ; ν C–OH
–	2941 (s)	2940 (m)	–	ν_{as} –CH ₃ ; ν C–OH
2870 (s, br)	–	2859(s, br)	–	ν_{s} –CH ₃ ; ν C–OH
2696 (m)	–	2722 (m)	2783 (w)	Overtone and combination bands
2666 (m)	2600 (w)	–	2643 (w)	
2587 (m)	–	2594 (m)	–	
2546 (m)	–	2534 (m)	–	
1755 (vvs)	1751 (m)	1748 (w)	1745 (sh)	
1692 (vvs)	1694 (w)	1685(sh)	1687 (w)	ν C=O (acid)
1605 (vs)	1606 (vvs)	1611 (vs)	–	ν C=C
1576 (w)	1576 (w)	1578 (m)	1582(m)	ν C–C
1518 (w)	–	1528 (w)	–	ν C=C
1483 (w)	1483 (w)	1483 (s)	1472 (s)	δ_{as} –CH ₃
1418 (s)	–	1443 (vvs)	–	β O–H
1369 (m)	1367 (w)	1383 (m)	1388 (m)	δ_{s} –CH ₃
1306 (vs)	1293 (m)	1296 (vs)	–	ν C–O (acid)
–	–	1248 (vs)	1246 (vs)	ν_{as} (ClO ₃)
1219 (s)	1223 (w)	1209 (s)	1206 (vw)	β C–H; ν C–O (acid)
1186 (vvs)	1191 (s)	1190 (sh)	1190 (vw)	β C–H
–	1154 (m)	1152 (m)	1153 (m)	β C–H
1092 (m)	–	1088 (w)	1093 (w)	β C–H; ν C–O (ester)
–	–	1069 (sh)	1068 (w)	ν_{s} (ClO ₃)
1038 (w)	1045 (m)	1042 (m)	–	β C–H; Ring breathing
1013 (m)	1014 (m)	1028 (w)	1030 (vs)	β C–H ; ν C–O (ester)
918 (s)	919 (w)	964 (w)	–	γ O–H
839 (m)	837 (w)	855 (m)	–	γ C–H
799 (m)	785 (m)	783 (m)	772 (vs)	γ C–H
754 (m)	751 (m)	758 (vs)	–	γ C–H
704 (m)	705 (m)	696 (vs)	–	γ C–H
–	–	658 (s)	663 (vw)	ν (Cl –OH)
–	–	615 (sh)	–	δ CO ₂ ; δ_{s} (ClO ₃)
563 (m)	551(m)	567 (m)	567 (m)	ρ CO ₂
–	–	463 (w)	452 (m)	ρ (ClO ₃)

s– strong; vvs- very very strong; vs- very strong; m– medium; w–weak; vw- very weak; sh– shoulder; ν – stretching; ν_{s} – sym. stretching; ν_{as} – asym. stretching; ρ – rocking; γ – out-of–plane bending; β – in–plane bending; δ_{as} – asym. bending; δ_{s} – sym. bending

The C–H out-of–plane (γ) and in–plane bending (β) occurs at 900 – 690 cm^{-1} and 1250–1000 cm^{-1} respectively [18, 20]. The wavenumbers at 839, 799, 754, 704 cm^{-1} in FT–IR spectrum and the wavenumbers at 837, 785, 751, 705 cm^{-1} in the Raman spectrum are identified as the γ (C–H) modes of pure aspirin. In the case of complex crystal this mode is identified at 855, 783, 758, 696 cm^{-1} and 772 cm^{-1} in the both spectra respectively. The C–H in–plane bending (β) modes of pure aspirin occurs at 1219, 1186, 1092, 1038, 1013 cm^{-1} in IR spectrum and at 1223, 1191, 1154, 1045, 1014 cm^{-1} in the Raman spectrum. The same mode is attributed to the complex crystal at 1209, 1190, 1152, 1088, 1042, 1028 cm^{-1} in the FT–IR and at 1153, 1093, 1030 cm^{-1} in the FT–Raman spectra

respectively. These modes agree well with the earlier reported values [17-20].

The ring carbon–carbon (C=C) stretching vibration occurs nearly in the region 1600 and 1500 cm^{-1} and is usually stronger [18]. These modes occur as two or three bands in the region due to skeletal vibration. In the present work, the C=C modes are observed experimentally as medium bands at 1605, 1518 cm^{-1} in FT–IR and at 1606 cm^{-1} in FT–Raman spectrum for pure aspirin crystal. For the complex crystal it is observed at 1611 cm^{-1} in FT–IR spectrum only and there is no its counterpart in the Raman spectrum for this mode. In the case of substituted benzene, the C–C stretching mode vibrations produce the bands at 1620–1565 cm^{-1} with the groups [13].

In the present compound, the bands at 1576 cm^{-1} in both spectra are assigned to C–C stretching vibration for pure aspirin crystal. These vibrational wavenumbers are very closely observed in the complex crystal of aspirin – perchloric acid. These results show that the benzene ring is not affected by the interaction of perchloric acid in the aspirin environment.

–CH₃ Group Vibrations

The –CH₃ stretching and deformation vibrations are more or less localized and give rise to good group wavenumbers. In aliphatic compounds the antisymmetric and symmetric –CH₃ stretching vibrations absorb near 2960 cm^{-1} and 2870 cm^{-1} respectively. Additional –CH₃ bands are also seen near 2934 cm^{-1} and 2912 cm^{-1} in some compounds [21]. These predictions hold well in the present study. The absorption bands of both crystals at $2999, 2940\text{ cm}^{-1}$ in IR spectra are attributed to antisymmetric –CH₃ stretching vibration. The same mode is observed at $2992, 2941\text{ cm}^{-1}$ in the Raman spectrum of pure aspirin only and Raman line is absent for complex crystal. Similarly the corresponding symmetric stretching mode of –CH₃ group is observed as strong bands at 2870 and 2859 cm^{-1} in IR spectrum of pure aspirin and aspirin-perchloric acid complex crystals respectively and there are no counterparts in the Raman spectrum for this mode.

The antisymmetric and symmetric deformation modes of –CH₃ group absorb nearly at 1465 cm^{-1} and 1378 cm^{-1} respectively [21]. In the present study, the bands at 1483 cm^{-1} , 1369 cm^{-1} and 1483 cm^{-1} , 1367 cm^{-1} in IR and Raman spectra respectively are attributed to antisymmetric and symmetric deformation modes of pure aspirin respectively. For the complex molecule these modes are observed at 1483 cm^{-1} , 1383 cm^{-1} and 1472 cm^{-1} , 1388 cm^{-1} in the IR and Raman spectra respectively.

HClO₄ Vibrations

Several investigators have studied IR and Raman spectra of perchloric acid. In this molecule, H atom is covalently bonded to one of the O atoms. Hence it is possible to consider the perchloric acid moiety as C_{3v} symmetry [22]. The OH stretching mode of HClO₄ appears in the infrared as a broad band peaking in the region of $3380\text{--}3340\text{ cm}^{-1}$ [22]. In the present compound the broad and weak bands that occur in the IR and Raman spectra of complex crystal at 3237 and 3233 cm^{-1} are assigned to the O–H stretching of perchloric acid. This vibration is found at the lower wavenumber side in both spectra of complex molecule due to the effect of hydrogen bonding in the condensed phase. The already reported vibrational wavenumbers of HClO₄ molecule are given as follows $\nu_{\text{as}}(\text{ClO}_3) = 1220\text{ cm}^{-1}$, $\nu_{\text{s}}(\text{ClO}_3) = 1039\text{ cm}^{-1}$, $\nu(\text{Cl–OH}) = 757\text{ cm}^{-1}$, $\delta_{\text{s}}(\text{ClO}_3) = 578\text{ cm}^{-1}$, $\rho(\text{ClO}_3) = 432\text{ cm}^{-1}$ [23, 24]. The assignment of bands corresponding to C_{3v} symmetry is presented below. The bands of ν_{asym} species of ClO₃ are identified at 1248 cm^{-1} and 1246 cm^{-1} in the IR and Raman spectra of complex molecule. The ν_{sym} mode of ClO₃ is identified in both spectra due to bands at 1069 and 1068 cm^{-1} respectively. These stretching wavenumbers are found to upshifting to higher wavenumber region due to the existence of covalent O–H bond associated with perchloric acid molecule. This leads to formation of strong bonding between the three O atoms with Cl atom. The band at 658 cm^{-1} in the IR and 663 cm^{-1} Raman spectrum of complex crystal is attributed to the $\nu(\text{Cl–OH})$ mode of HClO₄. The δ_{sym} mode of ClO₃ is observed as a shoulder band at 615 cm^{-1} in IR spectrum only. Finally the weak IR band at 463 cm^{-1} and medium Raman band at 452 cm^{-1} clearly helps to attribute for the rocking mode of ClO₃ moiety. These assignments agree well with the

earlier studies [23, 24]. These vibrational mode wavenumbers are not observed in the IR and Raman spectra of pure aspirin. This result strongly supports the incorporation of HClO₄ in the aspirin complex crystal.

UV–Visible Spectroscopy Analysis

The optical absorption spectra of pure aspirin and aspirin–perchloric acid crystals have been recorded with SHIMADZU–UV 1800 double beam spectrometer. Transmittance and absorbance data were observed for the crystals in the wavelength range $190\text{--}1100\text{ nm}$ in steps of 1 nm . The slit width chosen was 0.2 nm . The wavelength rate was in fast mode. The observed values of absorbance were recorded and stored in the memory of a computer and plotted. The absorption spectra of the pure aspirin and aspirin–perchloric acid complex crystals are shown in Fig. 7.

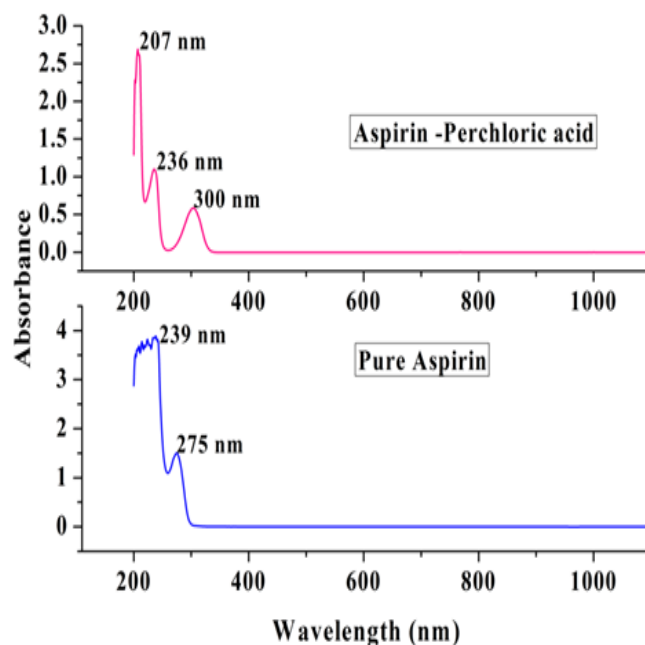


Figure 7: Absorption spectra of pure aspirin and aspirin–perchloric acid complex crystals.

From the absorption spectra, the both crystals show a good transmittance in the entire visible region. A good optical transmittance from ultraviolet to infrared region is very useful for optical applications. The lower cut–off wavelength is found to be 305 nm and 334 nm for pure aspirin and aspirin–perchloric acid complex crystals respectively. The optical window is found to be decreased in the presence of perchloric acid which is depicted in the fig.7. But there is no change in the transparency for both crystals in the visible region. The band gap of the crystal was estimated by using the following relation [25].

$$E_g = \frac{1.243 \times 10^3}{\lambda_{\text{max}}}$$

The band gap values of the crystals were found to be 4.0 eV (pure aspirin) and 3.72 eV (aspirin–perchloric acid). The band gap value of pure aspirin is decreased in the presence of the perchloric acid. This result also confirms the presence of perchloric acid in complex crystal.

SEM Analysis

The SEM analyzes were employed here to identify the surface morphology of the pure aspirin and aspirin–perchloric acid complex crystals. The crystals were cut into few mm sizes for observing the surface morphology. The SEM images of the pure aspirin and aspirin–perchloric acid complex crystals are shown in Fig. 8.

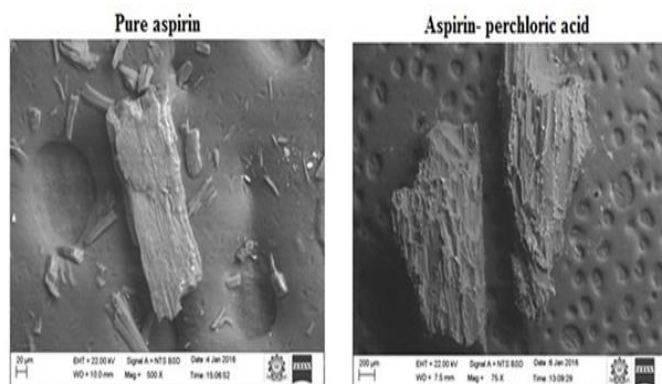


Figure 8: SEM images of pure aspirin and aspirin-perchloric acid complex crystals.

From the SEM micrographs, it can be seen that crystals have well defined shape with grooves. This shows the crystallite nature of the sample. The structural morphology of the pure aspirin consists of regular shape with grooves. But the shape of the complex crystal is entirely different from the pure aspirin with grooves.

Melting Point Analysis

The melting points of the pure aspirin and aspirin-perchloric acid crystals are determined by using the capillary tube method. Sometimes, this study is used to differentiate the pure sample from its complex form. Pure sample of aspirin usually have sharp melting point (135°C) while the HClO₄ doped compound melts at a lower temperature (110 °C). The melting points of the parent and the complex crystal are depicted in Table 3.

Table 3: Melting point of parent and the complex crystals.

Compound name	Melting point (°C)
Aspirin	135
Aspirin-Perchloric acid complex	110
Perchloric acid	-17

The melting point of the aspirin is decreased by the interaction of perchloric acid. This decreasing melting point confirms the formation of the aspirin –perchloric acid complex crystal.

Conclusions

A complex crystal of aspirin with perchloric acid is successfully grown by the vapor diffusion method. This complex crystal is investigated by employing powder XRD, FT-IR, FT-Raman, melting point, UV-Visible spectroscopy and SEM studies to confirm the formation of aspirin –perchloric acid complex crystal. The powder XRD study reveals the significant change of its X-ray diffraction peaks that might have resulted into the development of the complex between drug and perchloric acid. The FT-IR and FT-Raman spectra confirm the doping of perchloric acid with aspirin. From the UV-Visible absorption spectrum, the optical window is found to be decreased in the presence of perchloric acid. The band gap value of aspirin – perchloric acid complex is found to be as 3.72 eV. The surface morphology was also studied by SEM analysis and the results indicate that the complex crystal has different morphology from the pure aspirin crystal with the grooves. The melting point analysis also helps to differentiate the complex crystal from the parent. Finally, these studies conclude that the physicochemical

properties of pure aspirin drug have been changed by the incorporation of perchloric acid in the complex form. This property change is used to improve medicinal activity of drug compound in the biological systems.

Acknowledgements

The authors sincerely acknowledge their thanks to the Managements and Principals of Devanga Arts College, Aruppukottai and Thiagarajar College, Madurai for their permission and encouragement during this research work.

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