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SYNTHESIS, POWDER X-RAY DIFFRACTION PATTERN AND FTIR SPECTRUM OF POTASSIUM HYDROGEN PHTHALATE CHELATE ACETAMIDE

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ABSTRACT

Single crystals have been grown from an aqueous solution of potassium hydrogen phthalate enriched with equimolar concentration acetamide at room temperature. The powder XRD analysis reveals that the chelate crystals belongs to triclinic system with cell parameter α=7.81□, b=11.71□, c =6.32□, α=93.78°, β=102.76°, γ=94.86°. The coexistence of ghelate formation between KP and acetamide in the crystal confirmed by identifying the functional groups from FTIR spectrum. The grown crystal has UV absorption at 272 nm which indicate that the it is a potential candidate for NLO applications.

KEYWORDS

KHP, Acetamide and Chelate.

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INTRODUCTION

Potassium hydrogen phthalate (KHP) crystal is well known for its application in the production of crystal analyzer for long wave X-ray spectrometer. KHP possesses piezoelectric, pyroelectric, elastic and nonlinear optical properties. It crystallizes in orthorhombic structure with space group Pca21[1]. It has platelet morphology with perfect cleavages along (010) plane. Using the periodic bond chain analysis, the morphology of potassium hydrogen phthalate has been determined. Recently, KHP crystals are used as substrates for the growth of highly oriented film of conjugated polymers with

nonlinear optical susceptibility. KHP is chosen as a model compound because of its well-developed surface pattern on the (010) face consisting of high and very low growth steps which can be relatively easily observed by means of optical microscopy. The second harmonic generation (SHG) efficiency of sodium hydrogen phthalate is almost double that of KHP crystals[2]. The growth of KHP with several impurities has been undertaken by many researchers. Literature survey reports that organic impurities will influence the growth habit, surface phenomena and structural defects, as well as optical and physical properties of the crystals[3]. Acetamide (systematic name: ethanamide) is an organic compound with the formula CH_3CONH_2. It is the simplest amide derived from acetic acid. It finds some use as a plasticizer and as an industrial solvent[4]. The related compound *N*, *N*-dimethylacetamide (DMA) is more widely used, but it is not prepared from acetamide. Acetamide can be considered an intermediate between acetone, which has two methyl (CH_3) groups either side of the carbonyl (CO), and urea which has two amide (NH_2) groups in those locations[3]. The acetamide has two opposite polar groups: its carbonyl group (C=O) is a proton acceptor, which can easily interact electrostatically with the cation; its amino group (CH_2) is a proton donor and will interact with the anion. The electrostatic interaction between ions and the polar groups of acetamide occurs. One of the ions can electrostatically attract the polar group of the acetamide molecule which is opposite in polarity to the ion and make a negative contribution of the metal. The other will only interact structurally with a polar part of the acetamide[5]. Near the center of the Milky way galaxy, acetamide identified by researchers because it is potentially significant in finding the life in space. Researchers found the presence of acetamide on the Philae lander on comet 67/P's surface in July 2015. Because acetamide found infrequently on burning coal dumps as a mineral[6,7]. Based on these acetamide made to interact with KHP and its structural identification discussed in this work.

Experimental procedure

The chelate crystal was synthesized by dissolving the equi-molar concentrations (1:1) of AR grade KHP (E. Merck) and Acetamide in de-ionized water. The product was purified by recrystallization process using de-ionized water as a solvent. The crystals were grown by the slow evaporation solution growth technique. The transparent high-quality crystals harvested after 20 days. Photographs of the as-grown crystal are shown in Figure No.1.

MATERIAL AND METHODS

Powder XRAY diffraction pattern

X-ray powder diffraction was used to confirm single crystal XRD results. The efforts were made to record the powder XRD pattern of the crystal and index them. The comparison of the indexed powder XRD pattern of the grown crystal and KHP is shown in Figure No.8. Powder XRD pattern was recorded by scanning the crystal over the range 10-70° at a scan speed of 0.02°/min.

FTIR spectrum

FTIR spectrum data for the sample collected from the FT-IR spectrum of the synthesized compounds was measured in the 4000-400 cm^{-1} region using on SPECTRUM RX I spectrophotometer (Perkin Elmer).

UV-Vis spectrum

The absorption spectrum of synthesised compounds collected and has been recorded using UV–vis spectroscopy by employing a Systronics Double beam UV–vis spectrophotometer (Lambda 35, Perkin Elmer) operated on 190–1100 nm wavelengths.

RESULT AND DISCUSSION

Powder X-Ray diffraction pattern

The appearance of sharp and strong peaks confirms good crystalline nature of the grown crystal as shown in Figure No.2.

Cell parameter of Potassium hydrogen phthalate a=9.63□, b=13.34□, c=6.42 □, $\alpha = \beta = \gamma$=90°. The crystal has orthorhombic crystal system. Cell parameter of Acetamide Potassium Hydrogen

Phthalate a=7.81 □, b=11.71□, c=6.32□, α=93.78°, β=102.76°, γ=94.86°. The crystal was crystallized in triclinic system. The Scherrer formula t = 0.9 λ/ B cos θB is used to estimate the particle size of very small crystals from the measured width of their diffraction curves and are tabulated in Table No.1 and No.2. The comparison of the particle size shown in Figure No.3.

In potassium hydrogen phthalate crystals, the particle size varies as 2θ values increase. When Potassium Hydrogen Phthalate react with acetamide, the resultant crystal's particle size almost constant up to 2θ = 40° and above that there is an increase in the particle size.

FTIR spectrum of Potassium Hydrogen Phthalate chelate ac=etamide crystals

The FTIR spectrum of KHP is shown in Figure No.4, FTIR spectrum of acetamide shown in Figure No.5 and the FTIR spectrum of potassium hydrogen phthalate chelate acetamide crystals shown in Figure No.5.

In potassium hydrogen phthalate chelate acetamide crystal, the functional groups of KHP confirmed from the presence of C-C bond of aromatic ring indicated by the peak at 1563 cm^{-1}, the peak at 1484 cm^{-1} is due to the presence of O-C ring stretch, the peak at 1093 cm^{-1} due to the presence of C-C-O stretch, the peak at 851 cm^{-1} due to C-H out of plane, the peak at 440 cm^{-1} due to C-O wagging and the peak at 719 cm^{-1} due the presence of O-H out of plane bending.

The functional group of acetamide confirmed from the presence of NH_2 stretch at 550cm^{-1}, the amide identified from the peak at 1670 cm^{-1}, the presence of C=C identified from the peak at 1381 cm^{-1} and the CH_2 wagging occurs at 1281 cm^{-1}. The formation of chelate identified from the peak 2622 cm^{-1}.

UV spectrum

The UV absorption of KHP is at 300 nm and shown in Figure No.7. The UV absorption of acetamide is at 262 nm and is shown in Figure No.8. The UV absorption of potassium hydrogen phthalate chelate acetamide is at 272 nm shown in Figure No.9, which indicate that it may be the potential candidate for NLO studies.

Table No.1: Particle Size of Potassium Hydrogen Phthalate

S.No	2θ	FWHM	Calculated t (Å)
1	7.050	0.04	5.7
2	22.43	0.15	8.1
3	24.43	0.06	3.1
4	26.48	0.13	14.3
5	28.75	0.22	5.8
6	30.30	0.21	10.3
7	31.07	0.30	9.8
8	32.02	0.29	11.2
9	34.42	0.15	6.1
10	38.10	0.02	4.3

Table No.2: Particle Size of Potassium Hydrogen Phthalate chelate acetamide

S.No	2θ	FWHM	Calculated t (Å)
1	6.54	0.10	9.56
2	17.84	0.06	2.8
3	26.96	0.12	4.1
4	28.58	0.01	12.9
5	30.00	0.21	13.8
6	42.86	0.09	7.6
7	45.35	0.17	8.7
8	46.88	0.23	40.1
9	49.10	0.19	83.7
10	50.11	0.02	72.1

Table No.3: FTIR spectrum comparison of KHP, Acetamide and potassium hydrogen phthalate chelate acetamide

S.No	KHP		Acetamide		potassium hydrogen phthalate chelates acetamide	
	Frequency cm^{-1}	Vibration modes	Frequency cm^{-1}	Vibration modes	Frequency cm^{-1}	Vibration modes
1	1484	O-C ring stretching	1667	Amide group stretching	2622	Chelate
2	440	C-O wagging	1390	C=C	1563	C-C stretch aromatic ring
3	1562	C-C skeletal aromatic ring vibration	1308	CH_2 wagging	1484	O-C ring stretch
4	1088	C-C-O stretching	694	CH_3 wagging in plane	1093	C-C-O stretch
5	851	C-H out of plane bending	580	NH_2 stretch	851	C-H out of plane bending
6	1381	COO^- symmetric stretching			550	NH_2 stretching
7	719	O-H out of plane bending			440	C-O wagging
8					1670	Amide group stretch
9					1381	–C–C–
10					719	O-H out of plane bending
11					1281	CH_2 wagging

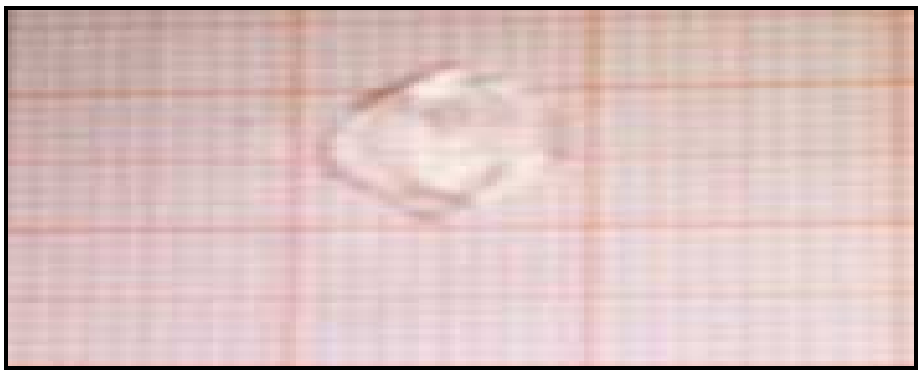

Figure No.1: Photograph of potassium hydrogen phthalate chelate acetamide crystal

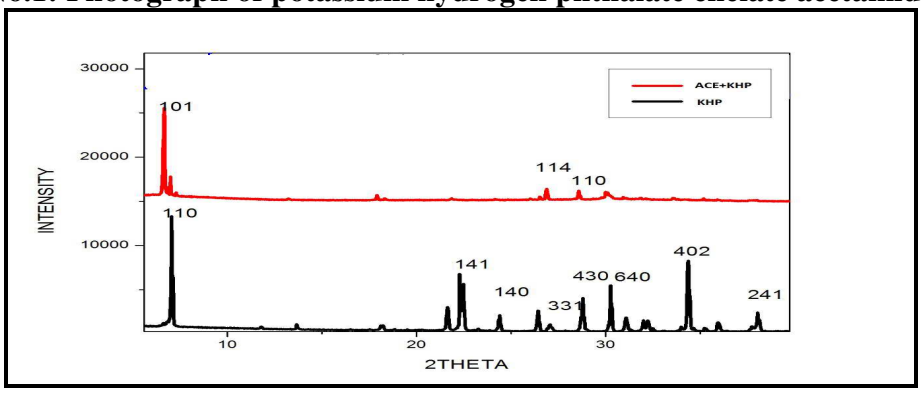

Figure No.2: Comparison of the powder X-Ray diffraction pattern between KHP and potassium hydrogen phthalate chelate acetamide crystals

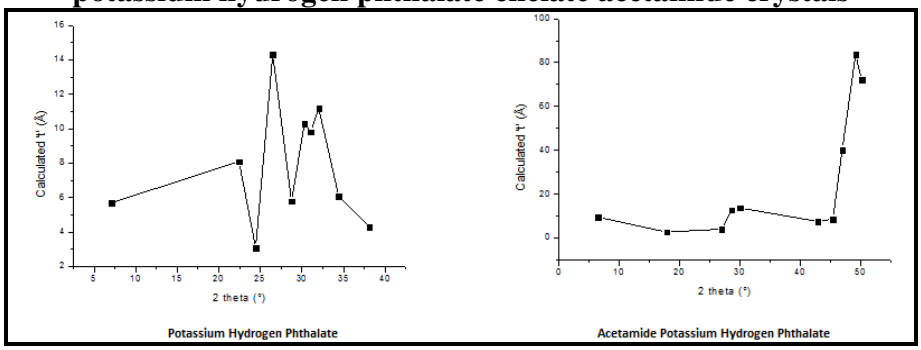

Figure No.3: Particle size comparison

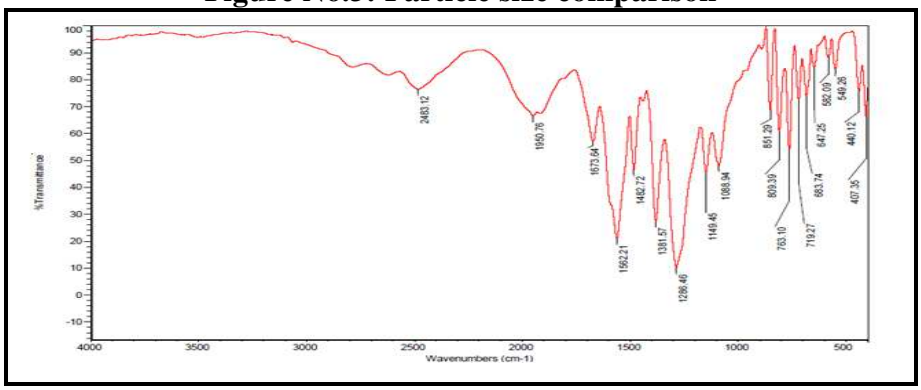

Figure No.4: FTIR spectrum of KHP

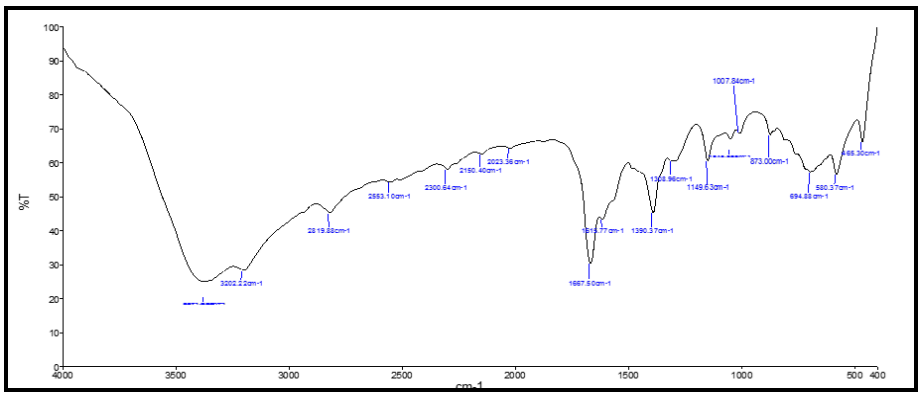

Figure No.5: FTIR spectrum of acetamide

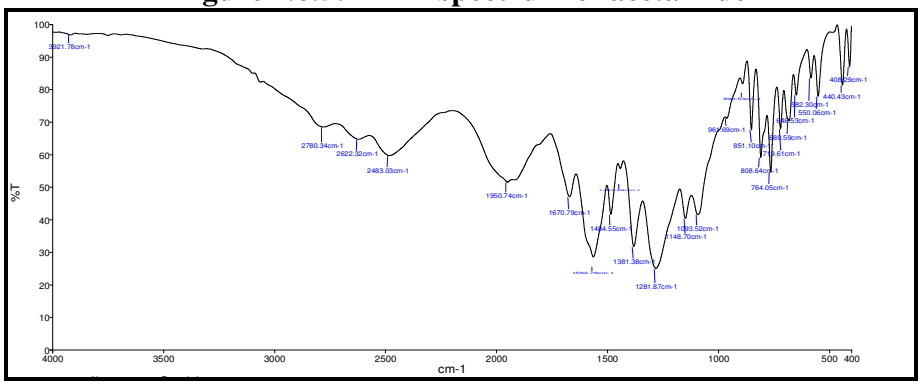

Figure No.6: FTIR spectrum of potassium hydro*gen phthalate chelate acetamide* crystals

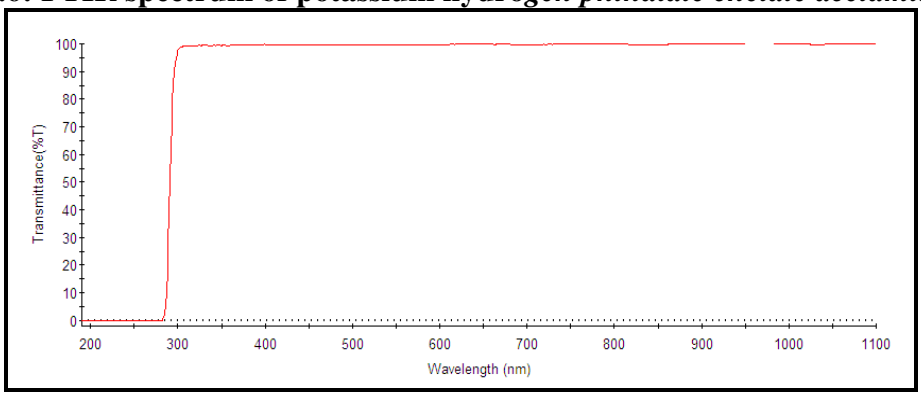

Figure No.7: UV spectrum of KHP

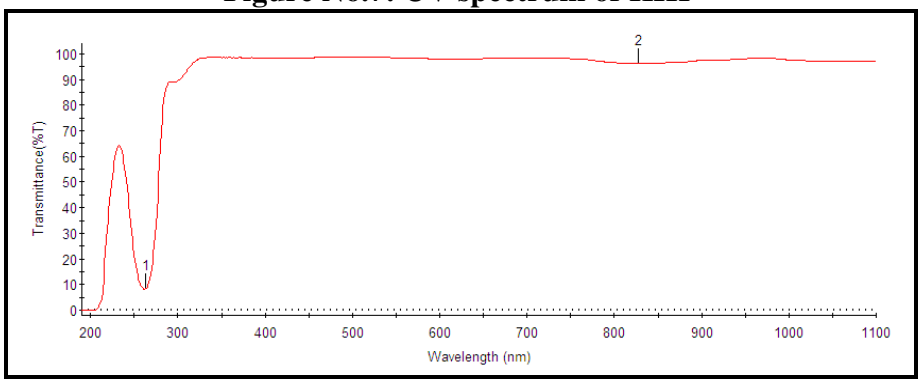

Figure No.8: UV spectrum of acetamide

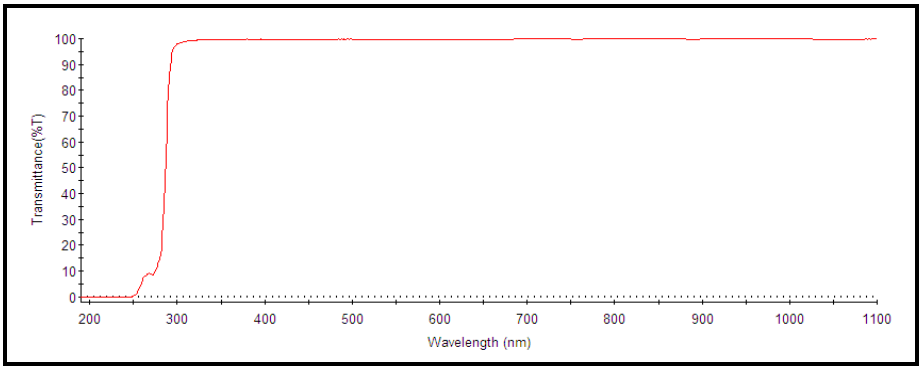

Figure No.9: UV spectrum of potassium hydrogen phthalate chelate acetamide

CONCLUSION

A new potassium hydrogen phthalate chelates acetamide single crystal synthesised and grown from the aqueous solution by slow evaporation. The particle size determined, and crystalline nature confirmed from the powder X-Ray diffraction pattern. The functional groups and the chelate formation between KHP and acetamide obtained from the FTIR spectrum. The UV absorption behaviour of the synthesised sample studied from the UV spectrum.

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CONFLICT OF INTEREST

We declare that we have no conflict of interest.

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