

Tunable Light Emission from P-Acetylbiphenyl-Cl-DPQ Organic Phosphor: For Blue and Near UV-OLED Applications

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Abstract. A novel 2-([1, 1'-biphenyl]-4-yl)-6-chloro-4-phenylquinoline (P-Acetylbiphenyl-Cl-DPQ) phosphor was synthesized by Friedlander condensation reaction at 140°C. To check its suitability for fabricating near UV – organic light emitting diodes (UV-OLEDs) by vacuum vapor deposition, the synthesized organic phosphor is assessed by various characterization techniques such as Fourier Transform Infrared (FTIR) spectroscopy, H-NMR, C-NMR, Thermo gravimetric/ Differential Thermal Analysis (TGA/DTA), optical absorption spectra and photoluminescence (PL) spectra in solid state. The synthesized phosphor is molecularly doped in Polystyrene matrix different wt% to explore its compatibility with polymers. FTIR and NMR confirm the structural formation of the desired complex. TGA and DTA reveal appreciable thermal stability and melting point. Absorption spectra were employed to study optical transitions in the synthesized phosphor at 10⁻³ M concentration in various organic solvents. The energy band gap of the synthesized organic phosphor was found to be 3.37, 3.40, 3.02, and 3.03 eV in chloroform, dichloromethane, acetic acid and formic acid, respectively. PL spectra of the phosphor in solid state, various organic solvents and in polystyrene reveal emission in the range of 383-495 nm, under excitation wavelength ranging between 360-419 nm. Stokes shift of about 63-66 nm was observed. CIE coordinates reflect that the synthesized organic phosphor can be used as a tunable emissive material in the range of blue to near UV emission in the fabrication of blue and near UV -OLEDs.

Keywords: Friedlander condensation reaction, Vacuum vapor deposition, solution techniques, near UV-LEDs, Solid state lighting.

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1 Introduction

In this era of world-wide expansion of opto-electronics, UV and near-UV emitters play a vital role. There are hardly few emitters with exceptional properties that demonstrate to be good UV-emitters.¹⁻⁴ Researchers are intriguing enormous efforts to expand organic materials that emit visible light.⁵⁻¹¹ Tremendous significance has been focused towards the organic systems that emit light in the broad range, from deep-blue to violet/ ultra violet region. As an ultimate output of research investigations, Ultraviolet (UV) to deep blue OLEDs were found have versatile applications in chemical and biological sensors displays.¹²⁻¹⁶ It is well known fact that shorter wavelength emission leads to lower device efficiencies but in recent times, there had been numerous reports¹⁷⁻²² of high efficiency blue-to-violet OLEDs which emit light in-between 400-480 nm with external quantum efficiency values up to 3% to 6%.²³⁻²⁶ UV OLEDs have relatively broad emission peaks, with emission tail extending to the longer wavelength ranging between 450-550 nm. There also exists some blue-violet-UV OLEDs in the prior state of art,

having emission peaking in the range of 374 - 443 nm.²⁷⁻²⁸ Regardless of various deep blue to near UV emitting small molecule OLEDs and polymer OLEDs,²⁹⁻³¹ only a few UV emitters reached up to 3% efficiency. There are two major challenges in the fabrication of UV OLEDs: (i) designing organic materials at molecular level capable of efficient UV emission and (ii) proposing an appropriate device structure,³²⁻³³ where both hole and electron carriers can be penetrated into a wide energy band gap of UV emitting materials. In this regard, we propose a novel design in the phosphor at molecular level, which is capable of manifesting tunable emission from blue to near UV region. This paper details (i) the synthesis of 1P-Acetyl-biphenyl-Cl-DPQ organic phosphor by Friedlander condensation reaction, (ii) structural, thermal and optical characterization of this new P-Acetylbiphenyl-Cl-DPQ with a ray of hope that this study could certainly ascertain good organization between their molecular structures and optical properties, which could afford an approach in designing realistically novel light emitting materials for blue and near UV- OLEDs.

2 Parts of a Manuscript

The synthesis of P-Acetylbiphenyl-Cl-DPQ was carried out in an inert atmosphere.

2.1 Reagent and Solvents

Materials used for the synthesis of 2-([1, 1'-biphenyl]-4-yl)-6-chloro-4-phenylquinoline (P-Acetylbiphenyl-Cl-DPQ) ($C_{27}H_{18}ClN$) phosphor are 4-Acetylbiphenyl ($C_{14}H_{12}O$) [Himedia] Molecular weight = 196.24, Minimum Assay 98%, 2-Amino-5-Chloro Benzophenone ($C_{13}H_{10}ClNO$) [Himedia] melting range 96-100⁰C, Molecular weight = 231.68 g/mol, Minimum Assay 98%, Diphenylphosphate (C_6H_5O)₂P(O)OH [Sigma Aldrich] 99% purity, melting point 62-66⁰C(lit.) Molecular weight = 250.19, m-cresol [$CH_3C_6H_4OH$] Molecular weight 108.14, 98.0% purity, Dichloromethane(CH_2Cl_2) [Fisher scientific] 99% purity, Wt. Per ml at 20⁰C 1.324-1.326g, Sodium hydroxide (NaOH) [Fisher scientific] Molecular weight 40.0, 98.0% purity, Hexane ($CH_3(CH_2)_4CH_3$) [Loba chemie] Molecular weight 86.18, 85.0% purity, Wt. Per ml at 20⁰C 0.66g and double distilled water. Chloroform ($CHCl_3$), [Fisher scientific] Molecular weight 119.38, 99.7% purity, Polystyrene [$CH_2CH(C_6H_5)$]_n, [Sigma Aldrich], Melting point ~ 240⁰C.

2.2 Synthesis of 2-([1, 1'-biphenyl]-4-yl)-6-chloro-4-phenylquinoline

A mixture of 2-Amino-5-Chloro Benzophenone ($C_{13}H_{10}ClNO$), (2g), 4-Acetylbiphenyl ($C_{14}H_{12}O$) (2g), Diphenylphosphate (2g) and M-cresol of 3ml were used as the starting materials during the synthesis process as shown in Fig.1(a). They were added into a three-neck flask with stirrer in the middle neck, which facilitates the process of stirring. The entire apparatus was inserted into an oil bath. Kept at constant temperature at about $90^{\circ}C$ for 1 hr and then at $140^{\circ}C$ for 4 hr. Purification was carried out by adding 60 ml of dichloromethane and 60 ml NaOH solution with 10 % NaOH concentration and the mixture was kept for 8 h. Funnel was used to separate the two layers, formed in the flask (Fig.1(b)). It was further washed with 50 ml of double distilled water.³⁴ Precipitate was then kept in an oven at $45^{\circ}C$ for 10 h and then washed with hexane again. The sample was kept in an oven at $40^{\circ}C$ for 6 hr. Finally, milky white color precipitate (2.56g) was obtained. 2D and 3D synthesis scheme for the synthesis of P-Acetylbiphenyl-Cl-DPQ are shown in Fig.2, respectively.

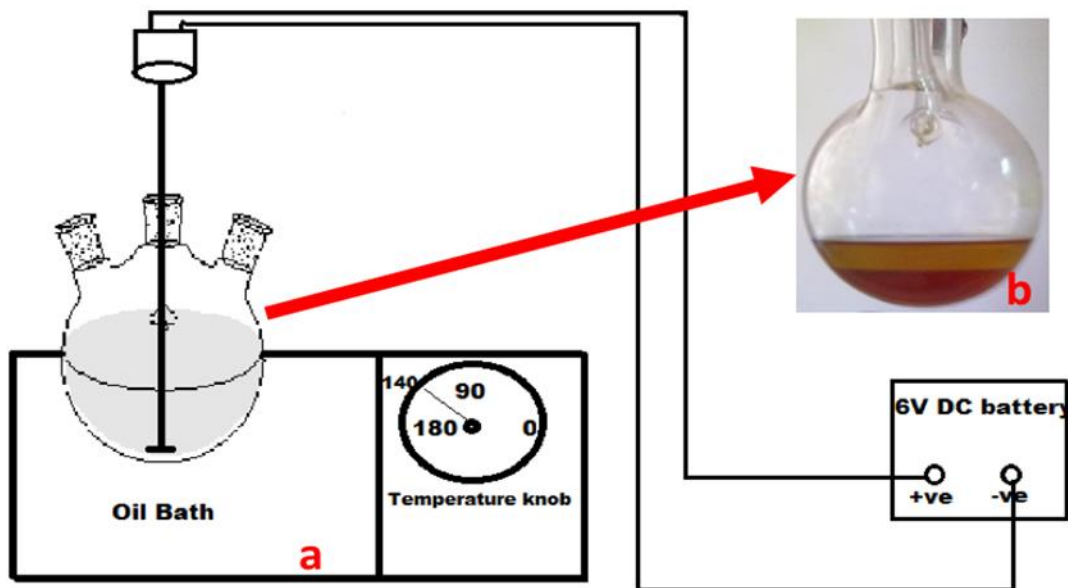


Fig.1 a) 2D-Synthesis scheme of P-Acetylbiphenyl-Cl-DPQ; b) Two layers formation during synthesis of P-Acetylbiphenyl-Cl-DPQ

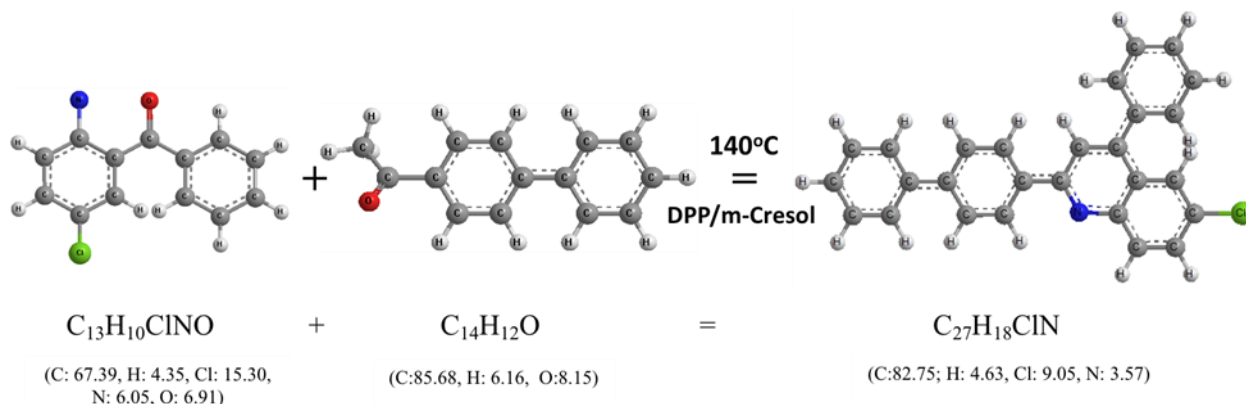


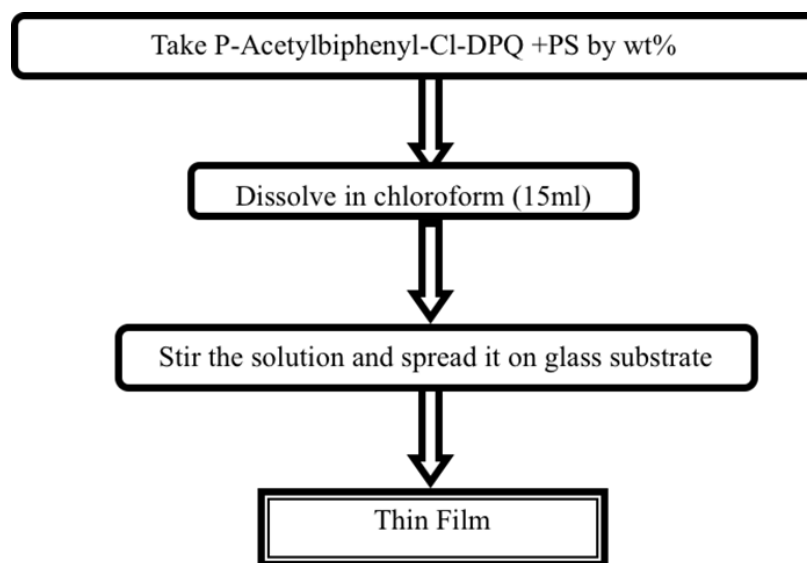
Fig.2 2D-Synthesis scheme of P-Acetylbiphenyl-Cl-DPQ

2.3 Preparation of blended films

In the present study, the compatibility of P-Acetylbiphenyl-Cl-DPQ in polystyrene (PS) is premeditated. It possesses high transparency, exhibits excellent thermal and electrical properties. It is chosen as a model polymer because it is also optically and electronically inert. Molecular structure and some important properties of polystyrene are displayed in Table 1. The blended films of small molecule organic phosphor were prepared by molecularly doping P-Acetylbiphenyl-Cl-DPQ in PS by wt%. These films were prepared by dissolving P-Acetylbiphenyl-Cl-DPQ and PS in chloroform (10, 5 and 1 wt %). Chloroform is selected as the solvent since the polymer PS with derivatives of DPQ are easily soluble. Good miscibility can be attributed to their structural similarities. The resulting homogeneous solution was casted on to a clean glass substrate for 10 to 15 minutes so that the solvent evaporates. Later, the films were taken out from the substrate carefully. The films were found to exhibit good transparency and homogeneity. All the films were dried overnight in a vacuum to remove any residual solvent. Photo physical properties of these films were then assessed by optical UV-vis absorption spectra and photoluminescence measurements. Preparation of blended thin films is schematically shown in Fig.3.

Chemical formula	$-[CH(C_6H_5)CH_2]_n-$
Synonyms	polystyrene PS
Color	Transparent
Density	1.04-1.065 g/cm ³

Melting point	240-250 °C
Glass transition	80-100 °C
Refractive index	1.59-1.60

Table 1 Properties of Polystyrene**Fig. 3** Schematic representation: Preparation of blended thin films

3 Result and Discussion

Structural properties of P-Acetylbiophenyl-Cl-DPQ are evaluated by Fourier Transform Infrared spectra (FTIR) on Bruker, H-NMR, C-NMR on Bruker Avance 400MHz, while thermal properties are explored by Perkin Elmer Diamond TGA/DTA analyzer. Optical absorption and emission spectra are carried out on HR 4C 4568 UV –Vis absorption spectra in acidic and basic media on Perkin Elmer LAMBDA 35 and Photoluminescence excitation spectra on Hamamatsu F-4500 RF5301 Spectro fluorometer, respectively. CIE coordinates were calculated on 1931 (Commission International d'Eclairage) system

3.1 FT-IR Spectra

FTIR was employed to confirm (i) the molecular structure (ii) absorption of a variety of IR light wavelengths in terms of wave number and (iii) the presence or absence of a wide variety of functional groups in P-Acetylbiophenyl-Cl-DPQ in-between $4000\text{--}400\text{ cm}^{-1}$ by averaging 500 scans at a maximum resolution of 20 cm^{-1} . It displays maximum absorption peaks in the range of

(600-1350 cm^{-1}), which can be assigned to intra molecular phenomenon as shown in Fig.4. Aromatic ring stretching was confirmed by a peak at 1352.43 cm^{-1} , while the peaks in-between 1000 – 600 cm^{-1} reveals the existence of phenyl group. Peaks at 692.10 cm^{-1} , 763.52 cm^{-1} and 833.59 cm^{-1} may be due to aromatic C-H vibration stretch. The aromatic C-C stretch bands were found to be prominent at 1477.58 cm^{-1} . The peak at 1676.86 cm^{-1} can be attributed to C=C bonding. Aromatic amines portray C-N stretch at 1260 cm^{-1} . These results confirm the presence of quinoline and the formation of P-Acetylbiphenyl-Cl-DPQ phosphor.

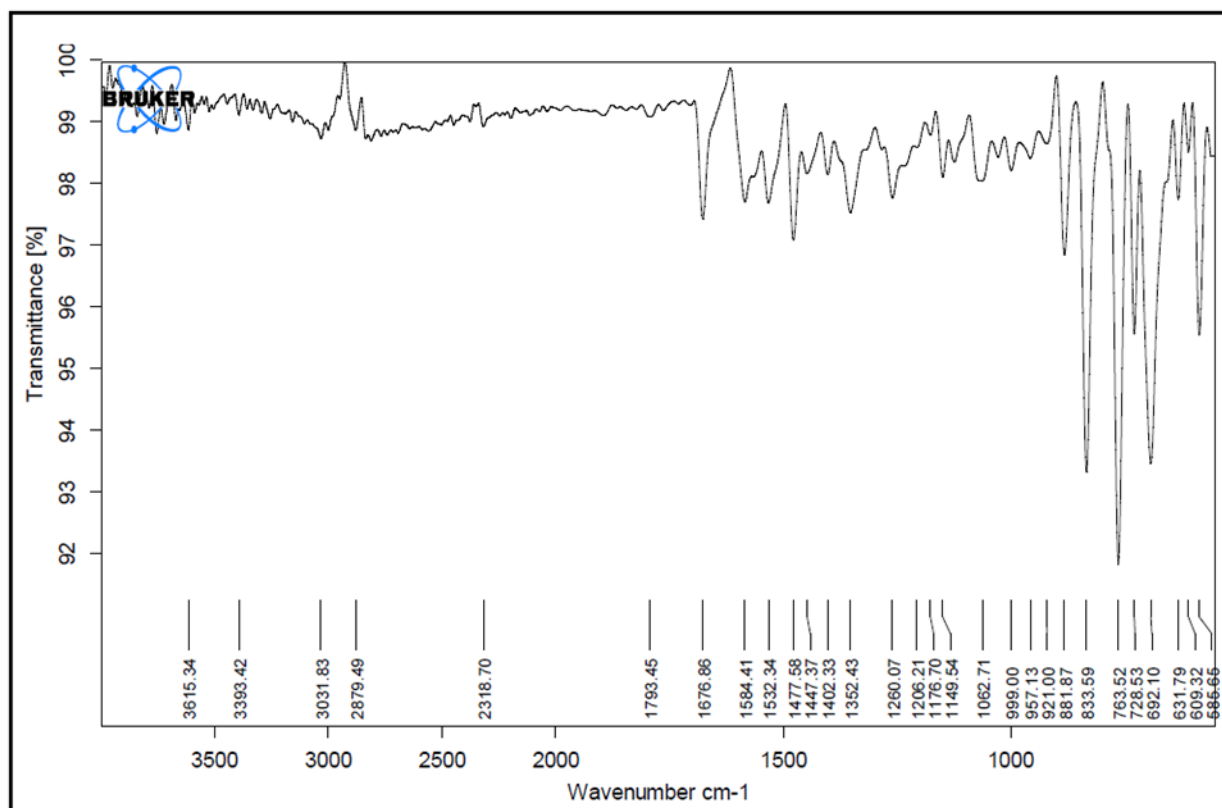


Fig. 4 FT-IR Spectra of P-Acetylbiphenyl-Cl-DPQ

3.2 NMR Spectra

NMR spectroscopy is an important tool to characterize the nature and the purity of the phosphor. Chemical shifts observed from the ^1H -NMR spectrum of P-Acetylbiphenyl-Cl-DPQ were found to be: H-NMR (400 MHz, chloroform- d (CDCl_3)) δ (ppm) 8.30 (d, 2H), 8.22 (d, 1H), 7.92 (s, 1H), 7.80 (m, 2H), 7.76 (m, 2H), 7.60 (m, 4H), 7.52 (m, 4H), 7.44 (m, 2H). Where d, s and m stand for doublet and singlet and multiplet bands, respectively as shown in Fig.5. This spectrum confirms the presence of 18 H-atom in the synthesized phosphor. These peaks can be assigned to the aromatic protons, which are correlated with the structure as described in Fig. 2. Chemical

shifts observed from the ^{13}C -NMR spectrum of P-Acetylbiphenyl-Cl-DPQ were found to be: ^{13}C NMR (100 MHz, CDCl_3) δ (ppm) 156.55, 148.48, 147.31, 142.28, 140.48, 138.05, 137.82, 132.25, 131.78, 130.52, 129.50, 128.96, 128.02, 127.77, 127.15, 126.53, 124.56, 119.54 as shown in Fig. 6. Each carbon nucleus has its own electronic environment and experiences different magnetic fields. P-Acetylbiphenyl-Cl-DPQ contains all 27 aromatic C-nuclei in the range of 115–160 ppm. Because of the symmetrically related nature of maximum C-nuclei, the ^{13}C spectrum displays 18 signals for a total number of 27 nuclei. The signals between 70 and 80 ppm are due to the chloroform.

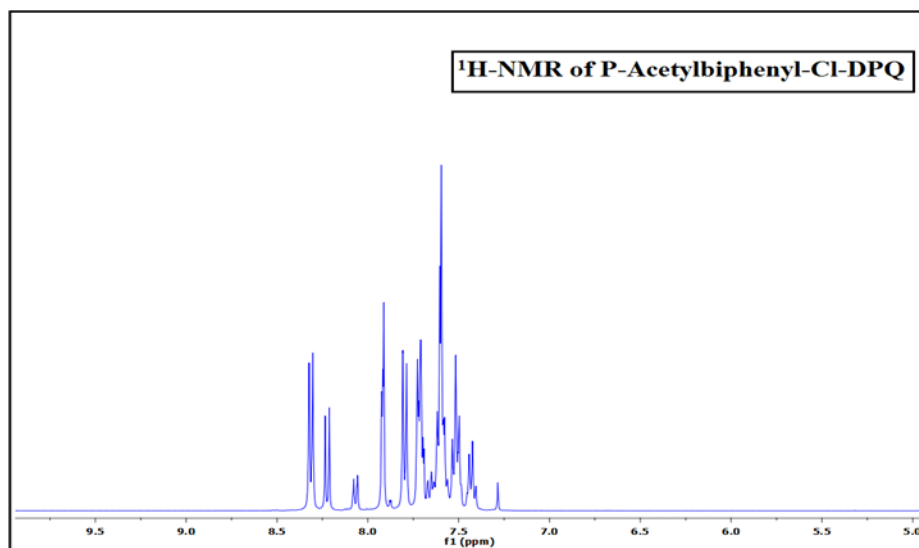


Fig.5 ^1H -NMR Spectra of P-Acetylbiphenyl-Cl-DPQ

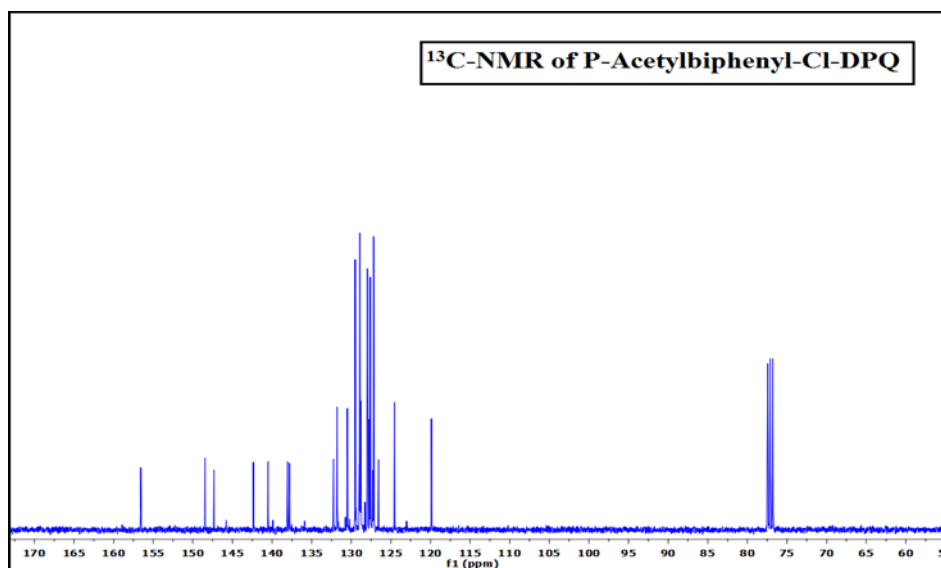


Fig.6 ^{13}C -NMR Spectra of P-Acetylbiphenyl-Cl-DPQ

3.4 UV- visible absorption spectra

In order to examine the shift of optical transitions in solvated P-Acetyl biphenyl-Cl-DPQ, in Chloroform, dichloromethane acetic acid, formic acid at 10^{-3} M, optical absorption spectra has been carried out. The solvated phosphor in chloroform and dichloromethane portrays peaks at 250, 342 nm and 235, 305 nm, respectively. Peaks at lower wavelength can be attributed to $\pi \rightarrow \pi^*$ transition of the conjugated polymer main chains, while peak at higher wavelength can be assigned to $n \rightarrow \pi^*$ optical transitions of conjugated side chains in DPQ moieties. Similarly, acetic acid and formic acid show absorption peaks at 284, 373 nm and 271, 373 nm, respectively with shift towards higher wavelength and decrease in optical intensity as shown in Fig.7.

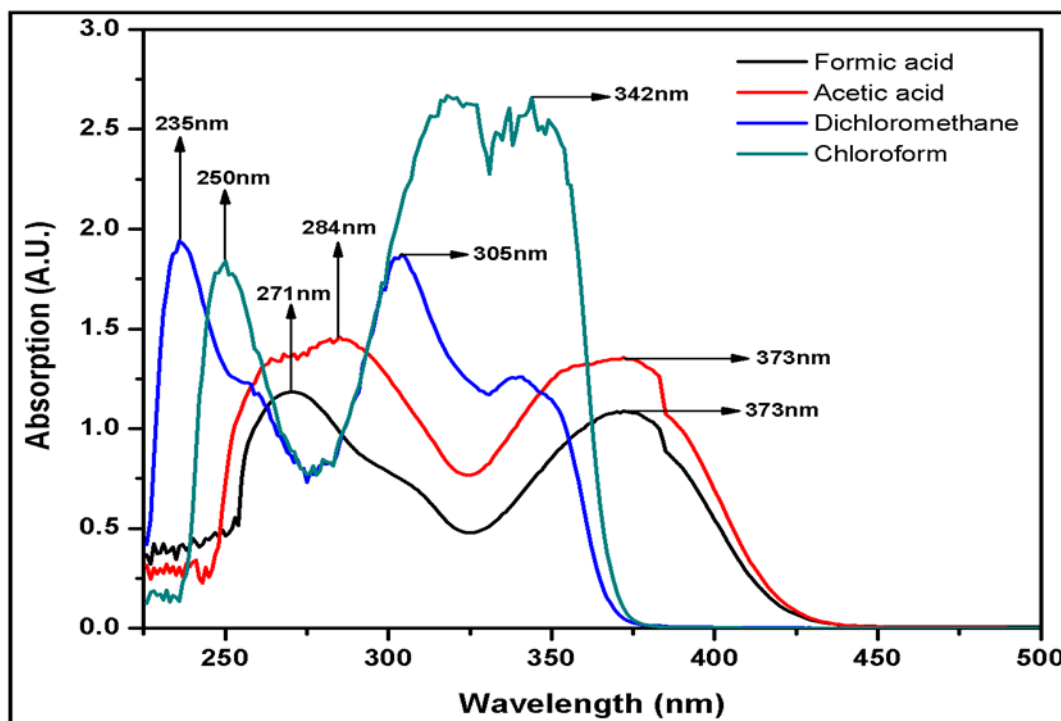


Fig. 7 UV-visible spectra of P-Acetyl biphenyl-Cl-DPQ in different solvents

Using the literature procedure,³⁵ the energy band gap of P-Acetyl biphenyl- Cl-DPQ was found to be 3.37, 3.40, 3.02, 3.03, eV in chloroform, dichloromethane, acetic acid and formic acid, respectively as shown in Fig.8

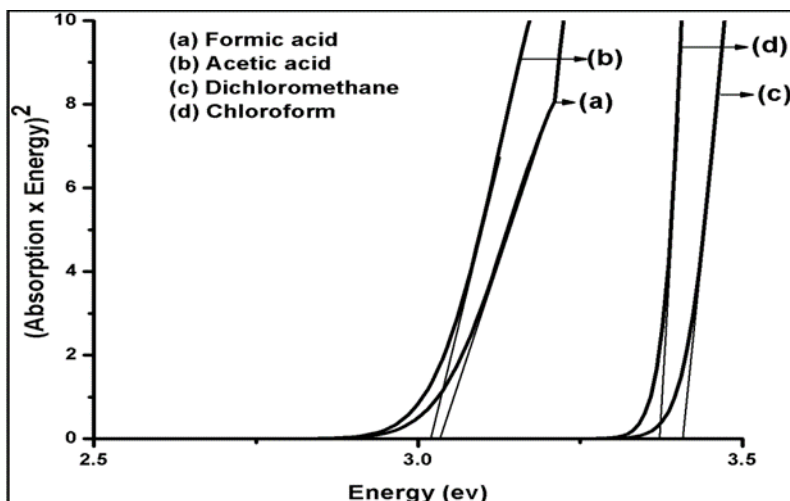


Fig. 8 Optical energy band gap of P-Acetylbiphenyl-Cl-DPQ in different solvents

3.5 Photoluminescence spectra

When excited under a wavelength of 377 nm, intense emission peak at 397 nm with few weak shoulder peaks, which falls in the near UV region of electromagnetic spectrum were observed as shown in Fig.9.

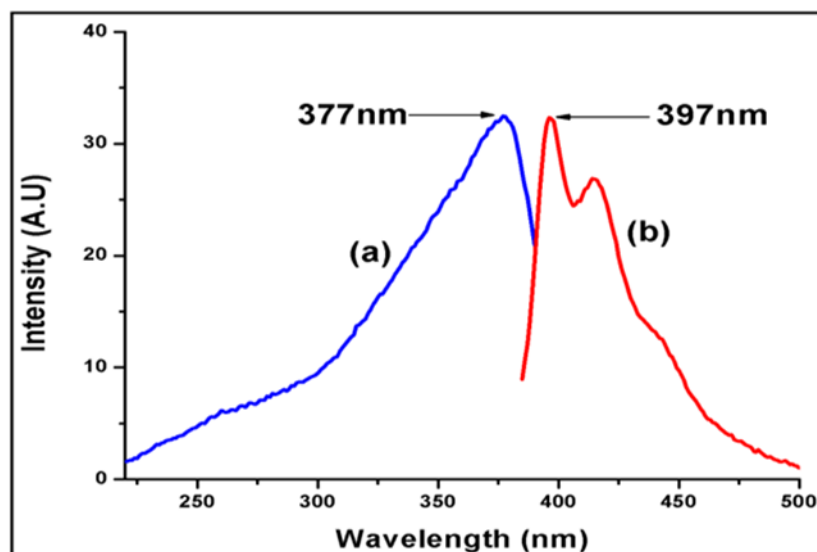


Fig.9 PL spectra of P-Acetylbiphenyl-Cl-DPQ

3.5.1 PL spectra of P-Acetylbiphenyl-Cl-DPQ in PS matrix

Fig.10 shows the excitation and emission spectra of 2-([1,1'-biphenyl]-4-yl)-6-chloro-4-phenylquinoline (P-Acetylbiphenyl-Cl-DPQ) + Polystyrene at 1, 5, 10wt %. Emission spectra for this series look similar upon excitation at a wavelength of 362 nm for 1 wt % and 366 nm for 5 and 10 wt%. The intensity of P-Acetylbiphenyl-Cl-DPQ was found to decrease with the

concentration and the position of λ_{emi} remained unaltered. However, change in the optical intensity, which depends on the concentration of P-Acetylbiphenyl-CI-DPQ was noticed. It may be attributed to poor interaction in-between P-Acetylbiphenyl-CI-DPQ phosphor and polystyrene at higher concentration.

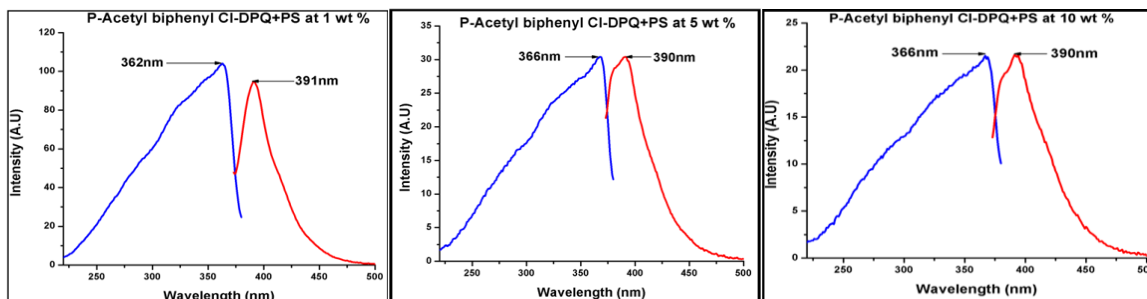


Fig. 10 PL spectra of P-Acetyl biphenyl-CI-DPQ+ Polystyrene for (a) 1wt% (b)5 wt% (c)10 wt%

3.5.2 PL spectra in different solvent

Fig.11 shows the photoluminescence (PL) spectra of solvated P-Acetylbiphenyl-CI-DPQ in various solvents like Chloroform (Non-polar), Dichloromethane (Polar aprotic) as well as different acids like acetic acid (polar protic) and formic acid (Polar- protic). The emission peak registered at 385, 383, 488 and 495 nm under excitation wavelength of 360, 362, 410 and 419nm, taken in the same order shown as fig. 12. The shift in excitation and emission wavelength can be attributed due to the changes in the polarity of solvent. The shift was found to be larger in protic solvents as compared to aprotic and non-polar solvents.

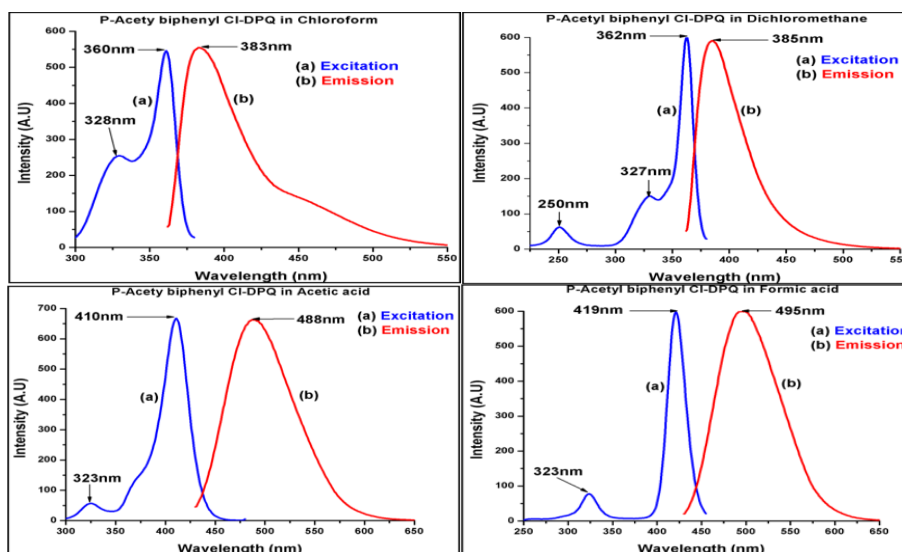


Fig.11 PL spectra of P-Acetylphenyl-CI-DPQ in (i) Chloroform (ii) Dichloromethane (iii) Acetic acid (iv) Formic acid

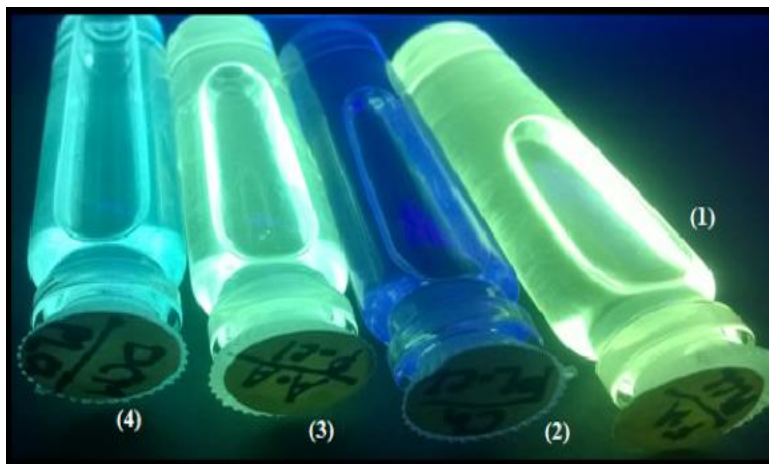


Fig.12 Color emission from P-Acetylphenyl-CI-DPQ in (i) Formic acid (2) Chloroform (3) Acetic acid and (4) Dichloromethane under UV light.

3.5.3 Stokes Shift

Fig.13 depicts the Stokes shift of P-Acetylphenyl-CI-DPQ in various solvents. Red shift (shift towards higher wavelength) is observed in formic acid as compared to acetic acid. The shift of emission in solution increased with increase in solvent polarity. The Stokes shift of P-Acetylphenyl-CI-DPQ was even larger [63- 66 nm], in protic solvents in comparison with aprotic and non-polar solvents. This increase in Stokes shift further support the view of intermolecular charge-transfer interactions in the phosphor.

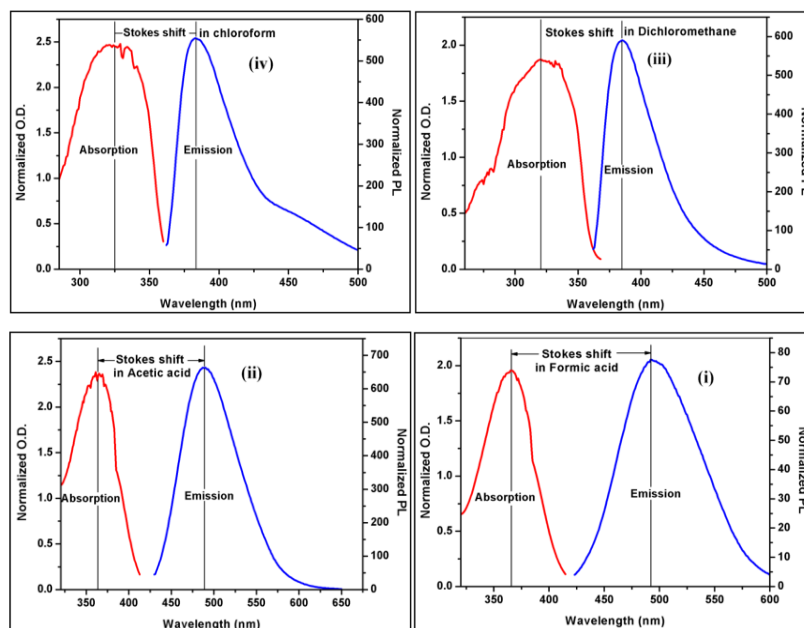


Fig.13 Stokes shift of P-Acetyl biphenyl Cl-DPQ in (i) Chloroform (ii) Dichloromethane (iii) Acetic acid (iv)Formic acid

3.5.4. CIE coordinates

CIE coordinates of P-Acetylbiphenyl-Cl-DPQ in various solvents were calculated from chromaticity coordinate calculation method based on the CIE 1931 (Commission International d'Eclairage) system.³⁶ The chromaticity coordinates shifted upon change in the solvent as shown in Fig.14. They were found to be (0.154, 0.072) in chloroform, (0.160, 0.040) in dichloromethane, (0.155, 0.352) in acetic acid and (0.172, 0.390) in formic acid. These results reflect the tunable light emission of the synthesized phosphor from blue to near UV region. Molar absorptivity was calculated by using Beer Lambert formula $A = \epsilon cl$. Table 2 demonstrates an exhaustive comparison of spectral properties of P-Acetylbiphenyl-Cl-DPQ in solid state, polymer matrix and in various solvents.

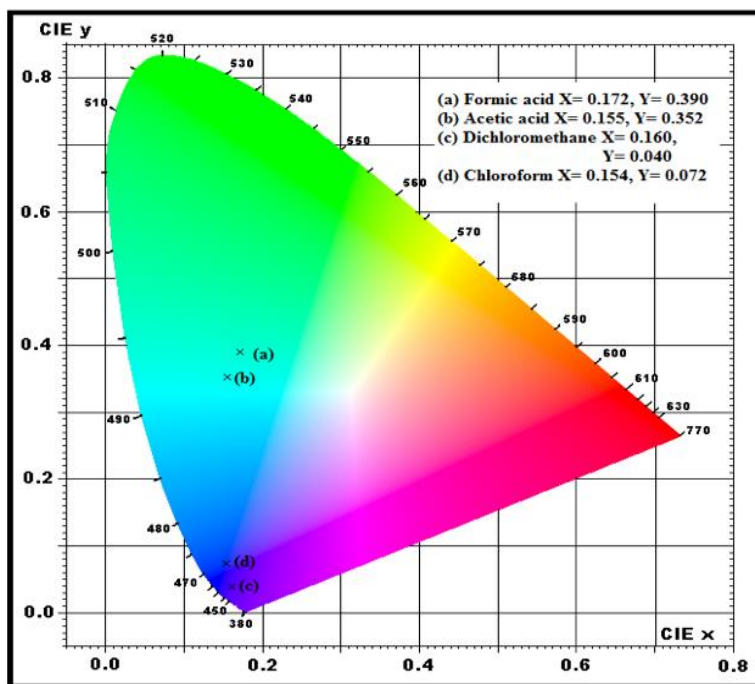


Fig.14 CIE 1931 (x, y) diagram of P- Acetylbiphenyl-Cl-DPQ in different solvents.

4 Conclusions

Novel P-Acetylbiphenyl-1Cl-DPQ was synthesized by Friedlander condensation reaction. The FTIR spectrum displays the molecular confirmation of P-Acetylbiphenyl-Cl-DPQ phosphor, ¹H-NMR spectrum of P-Acetylbiphenyl-Cl-DPQ confirms the presence of 18 H-atom in the synthesized phosphor. These peaks can be assigned to the aromatic protons, which were found to

be correlated with the structure. Chemical shifts observed from the ^{13}C -NMR spectrum of P-Acetylbiphenyl-Cl-DPQ depicts that each carbon nucleus has its own electronic environment and experiences different magnetic field. TGA and DTA reveal appreciable thermal stability. With the change in polarity index of the solvent, the absorption peak shifted towards long wavelengths, which may be due to protonation of the imine nitrogen of the quinoline ring to form the quinolinium ion. The energy band gap of the synthesized organic phosphor was found to be 3.37, 3.40, 3.02, and 3.03 eV in chloroform, dichloromethane, acetic acid and formic acid, respectively. When excited at 377 nm, the emission spectra of P-Acetylbiphenyl-Cl-DPQ in solid state display emission, peaking at 397 nm. The emission peak registered at 385, 383, 488 and 495 nm under excitation wavelength of 360, 362, 410 and 419 nm, taken in the same order. Emission spectra for this series look similar upon excitation at a wavelength of 362 nm for 1 wt. % and 366 nm for 5 wt.% and 10 wt.% with variations in optical density. Stokes shift of 63-66 nm was observed in solvated P-Acetylbiphenyl-Cl-DPQ. CIE diagram of the solvated phosphor displays coordinates of (0.154, 0.072) in chloroform, (0.160, 0.040) in dichloromethane, (0.155, 0.352) in acetic acid and (0.172, 0.390) in formic acid. These results reflect that the synthesized organic phosphor can be used as a tunable emissive material in the range of blue to near UV emission in the fabrication of blue and near UV -OLEDs.

Spectral properties of P Acetyl biphenyl Cl-DPQ in Solid state (Powder form)													
Solvent	Polarity Index	Absorption		Optical density		E _g (eV)	Molar absorptivity (ε)		Emission parameter		FWHM (nm)	Stokes shift (cm ⁻¹)	CIE Coordinate (x,y)
		Band 1 (λ _{max}) (nm)	Band 2 (λ _{max}) (nm)	Band 1 (a.u.)	Band 2 (a.u.)		Band 1	Band 2	λ _{ext} (nm)	λ _{ems} (nm)			
--	--	--	--	--	--	--	--	--	377	397	--	--	--
Spectral properties of P Acetyl biphenyl Cl-DPQ in Different solvents													
Formic acid	58	271	373	1.18	1.08	3.03	3.01	2.75	419	495	85	66	(0.172, 0.390)
Acetic acid	6.2	284	373	1.44	1.34	3.02	3.67	3.41	410	488	79	63	(0.155, 0.352)
Dichloromethane	9.1	235	304	1.88	1.87	3.40	4.79	4.77	362	385	46	69	(0.160, 0.040)
Chloroform	4.81	250	342	1.83	2.56	3.37	4.66	6.53	360	383	48	31	(0.154, 0.072)
Spectral properties of P Acetyl biphenyl Cl-DPQ in Blended film with PS													
10%	--	--	--	--	--	--	--	--	366	390	--	--	--
5%	--	--	--	--	--	--	--	--	366	390	--	--	--
1%	--	--	--	--	--	--	--	--	362	391	--	--	--

Table 2. Exhaustive comparison of spectral properties of 2-([1, 1'-biphenyl]-4-yl)-6-chloro-4-phenylquinoline (P Acetyl biphenyl Cl-DPQ) in solid state, polymer matrix and in various solvents.

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