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Absorption and Fluorescence Properties of 3,6-bis(5-bromothiophen-2-yl)-2,5-bis(2-ethylhexyl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione. A Covalent Fluorescence in Solution and in the Solid State

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Abstract:

In the current article, the photophysical properties of 3,6-bis(5-bromothiophen-2-yl)-2,5-bis(2-ethylhexyl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione were investigated. The visible absorption bands at 527, 558 and 362 nm in propylene carbonate and the compound was found to be fluorescent in solution and in the plastic film with emission wavelengths between 550- 750 nm. The Stokes Shift of P.C., acetonitrile, diethyl ether, Tetrahydrofuran THF, cyclohexane, dibutyl ether, and dichloromethane DCM are 734, 836, 668, 601, 601, 719, and 804 cm^{-1} in respectively. The Stokes Shift $\Delta\nu$ was less in THF and cyclohexane, than the solvents, which indicates that the energy loss is less between the excitation and fluorescence states. The photophysical characterizations were carried out on the compound in different solvents, the compound displays 12 nm red-shift in the absorption maximum in toluene compared with in propylene carbonate P.C, which the λ_{max} was 558 nm in P.C. The compound displays 5 and 10 red-shifts in the absorption maximum in diethyl ether and dibutyl ether respectively. The absorption band is almost insensitive to the polarity of the solvent, showing only a slight red shift from 563 nm in diethyl ether to 569 nm in cyclohexane $\Delta\lambda_{\text{max}}^{\text{Abs}} = 6 \text{ nm}$ and also red shift from 558 nm in acetonitrile to 570 nm in toluene $\Delta\lambda_{\text{max}}^{\text{Abs}} = 12 \text{ nm}$, which appreciably red-shifted depending on the polarity of the solvent. The emission maxima of the dye slightly shift more red region, such as DCM and toluene when compared this compound in other solvents. The dye showed a slight red shift *ca.* 5 nm on moving from the acetonitrile to the less polar solvent, such as dichloromethane DCM.

Keywords: Dihydropyrrolo, Intramolecular charge transfer, Plastic film, Red-shift, Stokes shift.

Introduction:

There are many applications in the field of fluorescence, for example the optoelectronic, organic light emitting diodes (OLEDs), organic photovoltaics (OPVs), organic light-emitting field transistors (LEFETs). The relationship between the molecular and film structure, and charge-transport and optical properties can be studied by using the new materials such as the small molecules, dendrimers, and polymers with optical and electronic properties. In OPV applications, the organic dyes or high electron affinity are used in these applications, when using the longer wavelength in absorption and low ionisation potential¹. The organic materials are very interesting molecules in science and industry, because of optoelectronic applications. The conjugated polymers are used for solar applications, which the

processes such as, exciton diffusion (ED)^{2,3}, charge transfer (CT)⁴⁻⁶, and excitation energy transport (EET)^{7,8} are used in the optical⁹ and electronic applications. In optoelectronic applications, the thiophene derivatives are used as active materials¹⁰⁻¹⁶. The chromophore is pyrrolo [3,4-c] pyrrole-1,4-dione-3,6-diyl (DPP) that is important and versatile compound for organic π -conjugated polymers, Fig. 1.

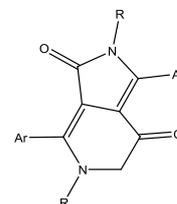
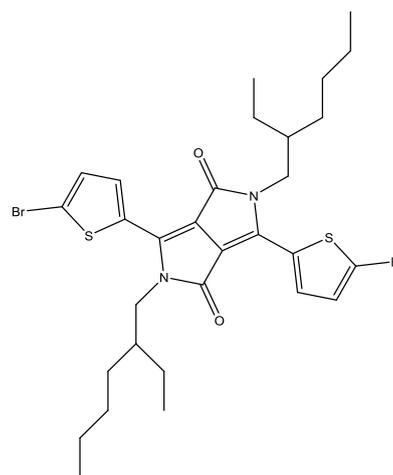


Figure 1. Chemical structure of pyrrolo [3,4-c] pyrrole-1,4-dione-3,6-diyl (DPP).

The π -systems are used in optoelectronic properties for example, in thin-film transistors and organic solar cells. The compound DPP with molecules are many good optical properties, for example tunable absorption wavelength, high extinction coefficients, and bright emission. The DPPs are bright fluorescence in solution, while many chromophores appear the photoluminescence activity¹⁷. The diketo-pyrrole-pyrroles (DPPs) are orange-red organic pigments, which are important in industry, the most pigments are 2,5-dihydropyrrolo[3,4-c] pyrrole-1,4-dione skeleton, this pigment consists of substituted phenyl rings, at positions 3 and 6 of the chromophore. The DPP derivatives consist of phenyl, that have many properties, for example high thermal stability, superior light fastness. The DPP derivatives are prepared to append different heterocyclic rings onto the chromophore of DPP core, and are used in many applications, such as organic semiconductor material for solar cells, gas sensing, and transistors for flexible electronics. Thiophene T or thienothiophene TT heterocycles are appended onto the chromophore of the DPP core. These materials are used as an organic semiconductors¹⁸. Farnum *et.al.* prepared the DPP chromophore in 1974, this molecule is an organic dye for ink industry and the painting. There are many excellent properties, for example high stabilities, high quantum yields for fluorescence, bright colours, high values of mobility and the activities of two-photoabsorption. The chromophore of DPP is a planar structure, and the coupling between the amide hydrogen atoms and neighbouring heteroatoms is observed by the strong intermolecular H-bonding, this is responsible for the red-shifts of the bands of absorption in the wavelength at 540 nm. The extending π -conjugation is responsible for improving the charge transfer and photon-electron ability of DPP dye¹⁹. DPP-based materials are strong fluorescence, therefore are used in biomedical applications, for example bio imaging and DNA elongation. The aggregation in solid states is observed in small molecular, DPP derivatives, for example thiophene-flankedDPP (1,4-diketo-3,6-di (thiophen-2-yl) pyrrolo[3,4c] pyrrole[DTDPP], by the strong intermolecular π - π interaction, the quenching of fluorescence happens by the aggregation²⁰. The DPP and their derivatives are used in many applications, for example paints, inks, organic photovoltaics, and fluorescent probes. The DPP-based conjugated polymers (CPs) are applied for photovoltaic or sensing applications²¹. DPP dyes are characterized by the strong fluorescence and moderate Stokes Shift SS, therefore these dyes are ideal molecules for the chemosensors design, bulk

heterojunction organic solar cells and solid-state lasers²². In bulk-heterojunction BHJ solar cells SCs contains the small molecules SM as a donor and the diketopyrrolo-pyrrole which is pyrrolo[3,4-c]pyrrole-1,4-dione DPP²³.

In the current work, the photophysical properties of the compound, 3,6-bis(5-bromothiophen-2-yl)-2,5-bis(2-ethylhexyl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione was reported, the UV- visible absorption and emission spectral properties were studied. The Stokes Shift ($\Delta\nu$) was calculated in different solvents, which is important for the fluorescent measurements. The structure of the compound was shown in Fig.2



3,6-bis(5-bromothiophen-2-yl)-2,5-bis(2-ethylhexyl)-2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione
Chemical Formula: C₃₀H₃₈Br₂N₂O₂S₂
Molecular Weight: 682.57

Figure 2. The structure of the compound.²³

The Experimental Part

Chemicals involve propylene carbonate, acetonitrile, diethyl ether, Tetrahydrofuran, cyclohexane, dibutyl ether, and dichloromethane.

Instrumentation absorption spectroscopy, the spectra were recorded using a Hitachi U3310 dual-beam spectrophotometer. Fluorescence spectra were recorded using a Hitachi F-4500 fluorescence spectrophotometer.

Results and Discussion:

The Absorption and Fluorescence Emission Spectra

The photophysical characterizations were carried out on the compound in different solvents, such as propylene carbonate, acetonitrile, toluene, dichloromethane, diethyl ether, cyclohexane, and dibutyl ether. It is to be noted that the solvents were selected based on the consideration that they can dissolve the compound. The absorption and fluorescence emission spectra are shown in (Figs. 3,4 and 5).The absorption spectra display a red shift²⁴ in the typical in some solvents, such as

toluene, dichloromethane, diethyl ether, cyclohexane, and dibutyl ether, compared with those in other solvents, such as propylene carbonate and acetonitrile. As for the compound displays 12 nm red-shift in the absorption maximum in toluene compared with in propylene carbonate P.C, which the λ_{max} was 558 nm in P.C. The compound displays 5 and 10 red-shifts in the absorption maximum in diethyl ether and dibutyl ether respectively. The intramolecular charge transfer ICT was found in the diketopyrrolo pyrrole DPP based-molecules, which the moiety is very electron-deficient and a strong acceptor²¹. The compound exhibits three characteristic absorption bands at 527, 558 and 362 nm in P.C., while four characteristic absorption bands at 570, 529, 356 and 371 nm in toluene.

Interestingly, the red-shift in λ_{max} value was not observed in more polar such as propylene carbonate and acetonitrile, which the λ_{max} is equal to 558 nm in P.C. and 558 nm in acetonitrile. The dye exhibits the lower polarizability of its excited state compared to the ground state in MeCN solution¹⁹. The absorption band is almost insensitive to the polarity of the solvent, showing only a slight red shift from 563 nm in diethyl ether to 569 nm in cyclohexane $\Delta\lambda_{max}^{Abs} = 6 \text{ nm}$ and also red shift from 558 nm in acetonitrile to 570 nm in toluene $\Delta\lambda_{max}^{Abs} = 12 \text{ nm}$, which appreciably red-shifted depending on the polarity of the solvent. As indicated in the absorption spectra, the dye has a common low-energy at 356 nm in some solvents, for example acetonitrile and toluene, which could be attributed to the lowest $\pi-\pi^*$ transitions. (Figure 4) shows the absorption of the dye after a pump with nitrogen gas.

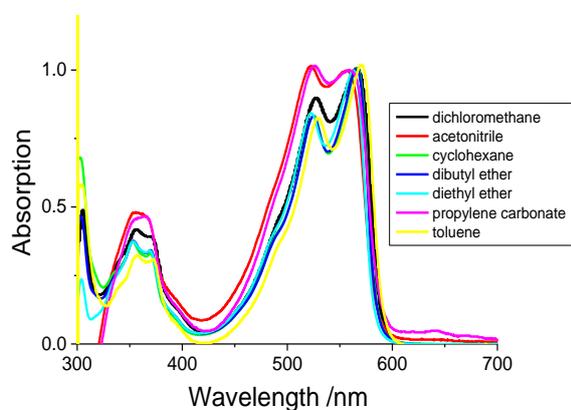


Figure 4. The absorption of the compound.

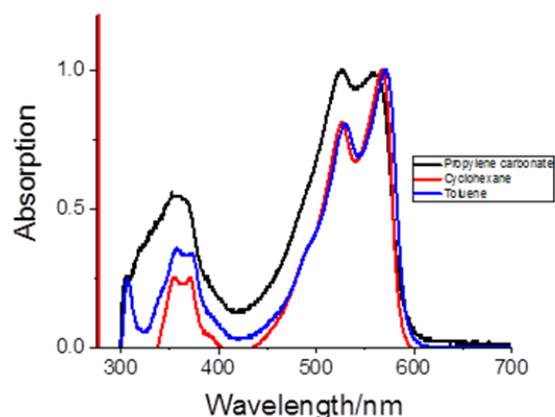


Figure 3. The absorption of the dye.

The fluorescence spectra of the dye are displayed in (Fig. 5). It can be observed that the compound shows mainly two emission bands, and the fluorescence maximum is changed according to the polarity of the used solvents. The emission maxima of the dye slightly shift more red region, such as DCM and toluene when compared this compound in other solvents. The dye showed a slight red shift *ca.* 5 nm on moving from the acetonitrile to the less polar solvent, such as dichloromethane DCM and a slight red shift *ca.* 7 nm on moving from acetonitrile to the less polar solvent, for example toluene. The emission of the dye follows the order Diethyl ether > acetonitrile > dibutyl ether > cyclohexane > P.C. > THF > DCM > and toluene. The compound showed a structured emission band displaying a slight red-shift from 583 nm in THF to 580 nm in acetonitrile, $\Delta\lambda_{max}^F = 3 \text{ nm}$. The emission maximum of the compound appreciably red-shifted from 579 nm in diethyl ether to 587 nm in toluene $\Delta\lambda_{max}^F = 8 \text{ nm}$.

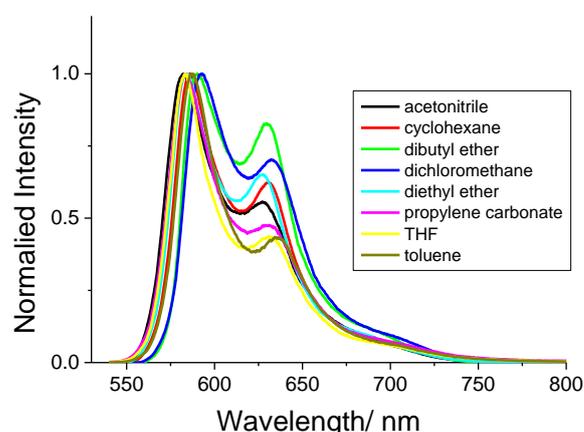


Figure 5. The fluorescence emission spectrum of the compound.

The Fluorescence of the Dye (Plastic Film) in CHCl₃

The emission spectrum is displayed in (Fig. 6), the peak maximum at 589 nm and also another peak at 637 nm these peaks are related to the n-π* and π-π* transitions. The film was slightly red-shifted by 9 nm and 6 nm compared with the dye in acetonitrile solvent, could be described by the intermolecular electronic interactions in the solid state, and the lower energy excited state stability²⁵. The fluorescence spectra after pumped the compound with nitrogen gas were measured in some solvent, for example P.C., cyclohexane, and toluene (Fig. 7). The film was slightly red-shifted by 9 nm and 6 nm compared with the dye in acetonitrile solvent, could be described by the intermolecular electronic interactions in the solid state.

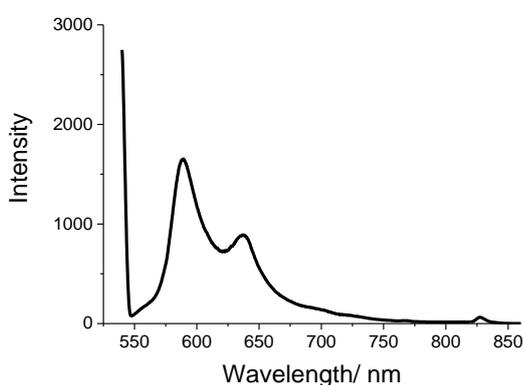


Figure 6. The fluorescence emission spectrum of the plastic film in CHCl₃.

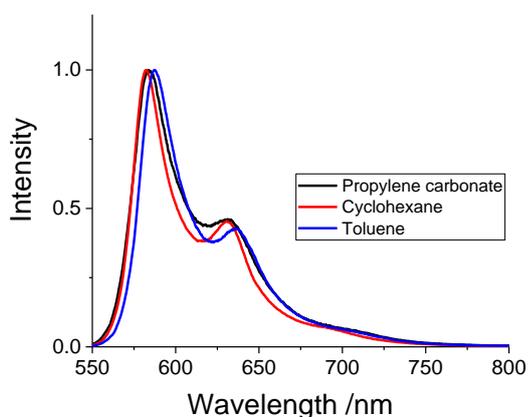


Figure 7. The fluorescence emission spectrum of the dye after a pump with nitrogen.

The Stokes Shift

The Stokes Shift²⁶ ($\Delta\nu$) is important for fluorescence measurements. The $\Delta\nu$ describes the difference in the structure between the ground state S₀ and the first excited state S₁. Equation 1 can be

described as the calculation of the Stokes Shift²⁷

$$\Delta\nu = \frac{\lambda_F - \lambda_A \times 10^7}{\lambda_F \lambda_A} \dots\dots\dots 1$$

Where λ_A and λ_F are wavelengths of the maximum absorption and fluorescence respectively. The $\Delta\nu$ was calculated for the dye in different solvents, the values are 734, 836, 668, 601, 601, 719, and 804 cm⁻¹ in P.C., acetonitrile, diethyl ether, THF, cyclohexane, dibutyl ether, and DCM respectively, (Figs. 8-14). The $\Delta\nu$ was less in THF and cyclohexane, than other solvents, which indicates the energy loss is less between the excitation and fluorescence states, therefore the molecules in the excited states are less vibrational relaxation or internal conversion in addition to the solvation effect²⁸.

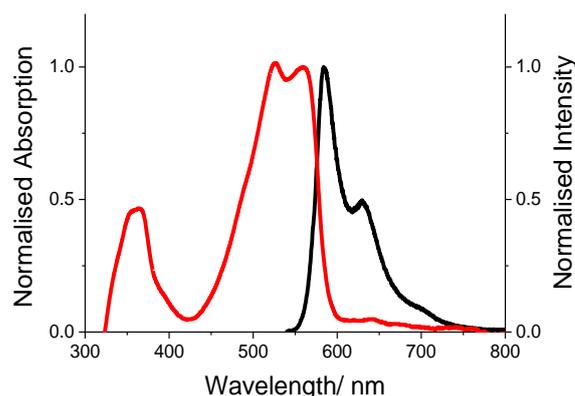


Figure 8. The absorption and the fluorescence spectra of the compound in propylene carbonate.

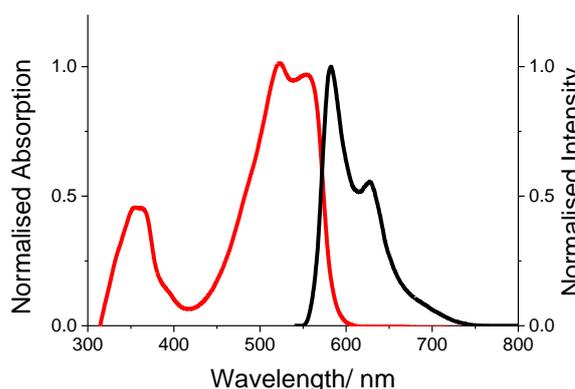


Figure 9. The absorption and the fluorescence spectra of the compound in acetonitrile.

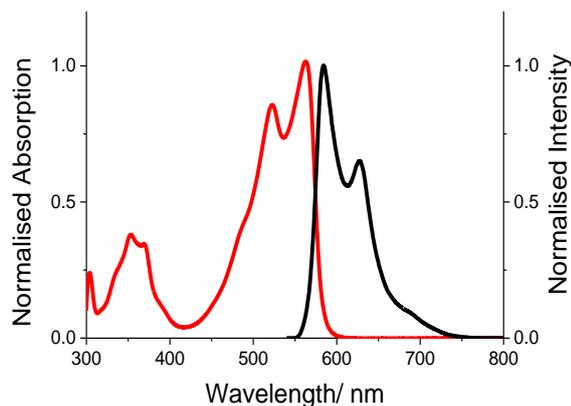


Figure 10. The absorption and the fluorescence spectra of the compound in diethylether.

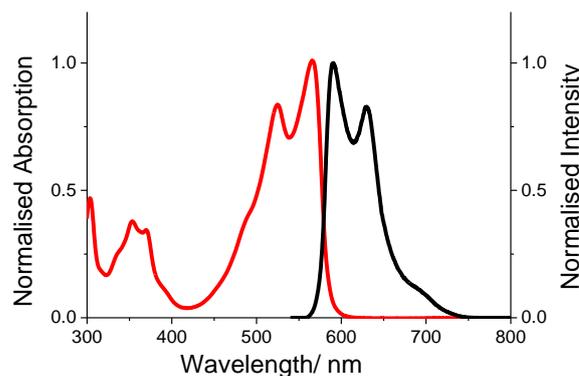


Figure 13. The absorption and the fluorescence spectra of the compound in dibutylether.

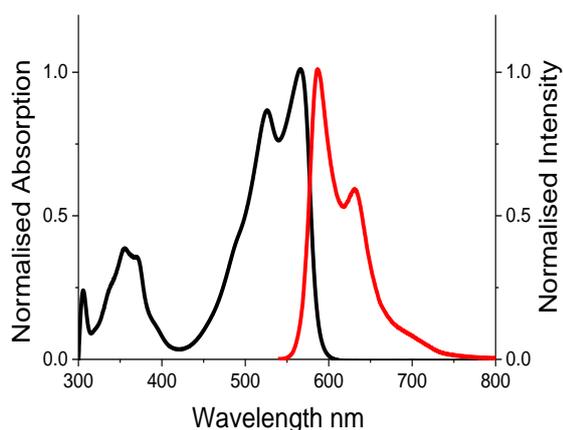


Figure 11. The absorption and the fluorescence spectra of the compound in THF.

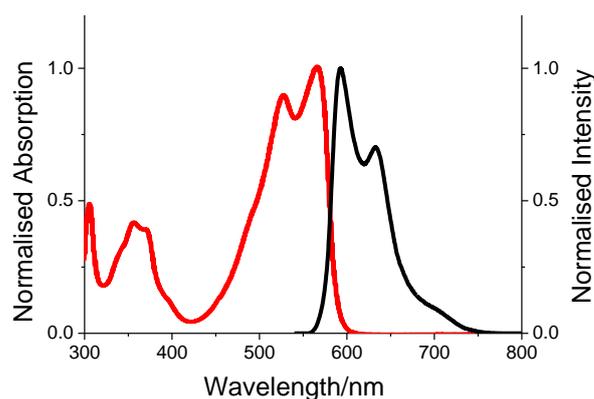


Figure 14. The absorption and the fluorescence spectra of the compound in DMC.

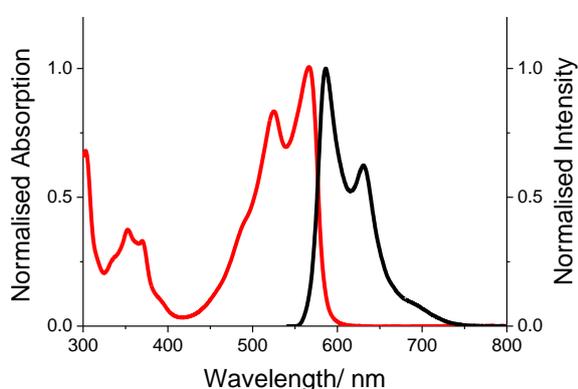


Figure 12. The absorption and the fluorescence spectra of the compound in cyclohexane.

Conclusions:

In summary, the photophysical characterizations were carried out on the compound in different solvents, the compound displays 12 nm red-shift in the absorption maximum in toluene compared with in propylene carbonate P.C, which the λ_{max} was 558 nm in P.C. The compound displays 5 and 10 red-shifts in the absorption maximum in diethyl ether and dibutyl ether respectively. The absorption band is almost insensitive to the polarity of the solvent, showing only a slight red shift from 563 nm in diethyl ether to 569 nm in cyclohexane $\Delta\lambda_{max}^{Abs} = 6\text{ nm}$ and also red shift from 558 nm in acetonitrile to 570 nm in toluene $\Delta\lambda_{max}^{Abs} = 12\text{ nm}$, which appreciably red-shifted depending on the polarity of the solvent. The emission maxima of the dye slightly shifted more red region, such as DCM and toluene when comparing this compound in other solvents. The dye showed a slight red shift *ca.* 5 nm on moving from the acetonitrile to the less polar solvent, such as dichloromethane DCM and a slight red shift *ca.* 7 nm on moving from acetonitrile to the less polar solvent, for example toluene. The Stokes Shift $\Delta\nu$ was less in THF and cyclohexane,

than other solvents, which indicates the energy loss is less between the excitation and fluorescence states. DPP dyes are characterized by the strong fluorescence and moderate Stokes Shift SS, therefore these dyes are ideal molecules for the chemosensors design, bulk heterojunction organic solar cells and solid-state lasers.

Acknowledgement

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Author's declaration:

- Conflicts of Interest: None.
- I hereby confirm that all the Figures and Tables in the manuscript are mine. Besides, the Figures and images, which are not mine, have been given the permission for re-publication attached with the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee in University of Newcastle.

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صفات التفلور والامتصاصية لمركب 6، 3- بس (5- برومو ثايوفينا-2-يل) – 2، 5- بس (2-ايتايل هكسايل) 2,5- داي هايدرو بايرونلو (3,4-سي) بايرونلو-1,4-دايون. التفلور في المحلول والحالة الصلبة

روزا عبدالرزاق صالح العقار

قسم الكيمياء، كلية العلوم، جامعة البصرة، البصرة، العراق

الخلاصة:

تم في هذا البحث دراسة الصفات الفيزيائية الضوئية لمركب 3,6 – بس (5- برومو ثايوفينا-2-يل) – 2، 5- بس (2-ايتايل هكسايل) 2,5- داي هايدرو بايرونلو (3,4-سي) بايرونلو-1,4-دايون eh433f1. تظهر حزم الامتصاصية في المنطقه المرئية عند 527,558، و 362 نانوميتر في مذبذب كربونات البروبلين وأيضا يظهر المركب تفلور في المحلول وفي فلم البلاستيك بمدى 750-550 نانوميتر. ان قيم الستوك شفت للمركب تساوي 734، 836، 668، 601، 601، 719، 804، سم⁻¹ في مذبذبات كربونات البروبلين، الاستيونايترايل، داي اثيل ايثر، تي اج اف ، سايكوهكسان، داي بيوتايل ايثر، دي سي ام على التوالي. يظهر الفلم ازاحه حمراء حوالي 9 و 6 نانوميتر مقارنة مع الصبغه في مذبذب الاستيونايترايل والذي يوصف نتيجة التداخلات الالكترونية داخل الجزيئه. ان قيم الستوك شفت تكون قليلة في مذبذبات التي اج اف والسايكلوهكسان مقارنة مع باقي المذبذبات والسبب يعود الى قلة فقدان الطاقة في حالات التفلور والاثاره. شخصت الصفات الفيزيائية الضوئية للمركب في المذبذبات المختلفه اذ يظهر ازاحه حمراء قدرها 12 نانوميتر في مذبذب التولوين مقارنة مع مذبذب كربونات البروبلين حيث ان قمه الطول الموجي 558 نانوميتر في مذبذب كربونات البروبلين. يظهر المركب ازاحه حمراء في قمه الامتصاصيه قدرها 5 و 10 نانوميتر في مذبذبات الداي اثيل ايثر والداي بيوتايل ايثر على التوالي. ان حزمه الامتصاص تكون غير حساسه الى قطبيه المذبذب أي تظهر ازاحه حمراء في 563 نانوميتر في داي اثيل ايثر الى 569 نانوميتر في السايكلوهكسان أي $\Delta\lambda_{max}^{Abs} = 6$ نانوميتر وايضا ازاحه حمراء في 558 نانوميتر في الاستيونايترايل الى 570 نانوميتر في التولوين $\Delta\lambda_{max}^{Abs} = 12$ نانوميتر، حيث تعتمد الازاحه الحمراء على قطبيه المذبذب. ان قمه حزمه الانبعاث للصبغه تظهر ازاحه حمراء في الذي سي ام والتولوين مقارنة مع المذبذبات الاخرى. تظهر الصبغه ازاحه حمراء قدرها 5 نانوميتر بالانتقال من مذبذب الاستيونايترايل الى مذبذب اقل قطبيه مثل داي كلورو ميثان الذي سي ام.

الكلمات المفتاحيه: داي هايدروبايرونلو، انتقال الشحنة ضمن الجزيئه، فلم البلاستيك، ازاحه حمراء، الستوك شفت.