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STRUCTURAL, OPTICAL, ELECTROCHEMICAL, THERMAL AND ELECTRICAL PROPERTIES OF 6, 13-BIS (TRI-ISOPROPYLSILYLETHYNYL) TIPS-PENTACENE

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We report on the small molecule of 6, 13-bis (tri-isopropylsilylethynyl) pentacene (TIPS-pentacene) powder and solution grown single crystal structural properties while the optical, electrochemical properties are studied by using TIPS-pentacene solution, thermal and electrical properties are studied by using its powder. TIPS-pentacene is preferable to other organic materials due to their remarkable electronic properties, stability and solubility. The studies performed by X-ray diffraction technique indicate that the material is single phase having a triclinic structure. The grown single crystals are highly oriented. From UV-visible spectroscopy analysis, it has been observed that the absorption peaks lie within the range of 300-643 nm. Thermal behavior has been studied by Thermo Gravimetric Analysis (TGA) and Differential Scanning Calorimeter (DSC) that the material decomposes at 412 ºC and melts at 263 ºC.

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Keywords: X-ray scattering; Thermo gravimetric analysis (TGA); Differential Scanning Calorimeter (DSC); Fourier transform infrared spectroscopy (FTIR).

1. Introduction

The interest in organic semiconductors based on conjugated materials has been emerged in the early 1980. Many organic solids, polymers are perfect insulators, rarely are conductors and have been an integral part of the semiconductor industry since 1960. The first metal oxide silicon field effect transistor is demonstrated by Kahng and Atalla [1]. Theinvention of conducting polymers becomes pioneer for the organic semiconducting materials. Organic semiconductors are more applicable than inorganic one due to their light weight and ease in processing. The conjugated polymers appear as a new class of organic materials with promising electrical properties. Conjugated polymers and small molecules are now a day's subject to intense research owing to their potential applications in photonic and optoelectronics [2-3]. TIPS- pentacene is a solution process able small molecule that forms Van der Waals bonded molecular system. TIPS pentacene small molecule is formed when tri-isopropylsilylethynyl acetylene is added to a pentacene. TIPS-pentacene molecules assume two-dimensional π-π stacking within unit cell. The presences of the isopropyl group make this material soluble in common organic solvent. The low cost, ease of fabrication and tunable properties make the organic semiconductor more preferable than inorganic semiconductor. Conductive polymers are light weight and inexpensive. These are widely used in the field of solid state such as batteries, electrochemical displays, electrochemical sensors [8, 9] and transistors [10-13]. At the same time functionalized pentacene can be readily processed from common organic solvents such as m-xylene, CCl_4, chloroform, bromobenzene, tetrahedrofuran and toluene [14]. Among the solution processing organic semiconductors, 6, 13-bis (tri-isopropylsilyethynyl) pentacene is a known well due to its high Hole mobility and environment stability. However, its tendency to form randomly oriented needle shaped crystals,

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when its solution is crystallized, leads to significant performance variations in organic thin film transistors performance. TIPS pentacene stacked in two dimensional columnar arrays, gave rise to an electronic structure that is different from pentacene. Such differences make a case for fundamental studies in OTFTs using TIPS pentacene as the semiconducting layer even though there have been several studies dedicated to pentacene based OFETs [15-19]. TIPS pentacene can be crystallized in the thin film form, leading to a strong bathochromic shift, which indicates strong π-π interactions. Since crystalline thin films absorb further into the red then un-substituted pentacene, this leads to higher photocurrent and power conversion efficiency [20]. Pentacene is one of the small organic molecules which have the highest charge carrier mobility. In comparison to pentacene 6, 13-bis (tri-isopropylsilyethynyl) pentacene is better in solubility and stability due to the presence of silyl groups. TIPS-pentacene, rubrene exhibits the highest mobility as a result these are commonly used in the organic field effect transistors and in the optoelectronic devices [21-24]. Many complex microstructural organic semiconductor materials tend to form films that have a great impact on device functionality. These materials are classified into two major groups, the small molecule based and polymer based organic semiconductor both having different microstructures. Small molecule based films can possess longer range order, regular arrangement of these small molecules within each domain enhances exciton and charge diffusion, improving device performance while Polymer based films are dissolved with small extent. TIPS pentacene marked the turning point in the field of organic thin film transistors as the molecule is easily synthesized in good yield, stable in normal conditions and exhibits carrier mobility comparable to and better than amorphous silicon. Recent research however expands the expertoire of semiconducting materials to include certain organic molecules. Such organic semiconductors are superior to their inorganic counterparts because they are easily process able to deposit on a substrate, light in weight and significantly more flexible. In this paper, we have described the structural, optical, thermal and electrical properties of TIPS-pentacene.

2. Experimental

In order to get single crystals of TIPS-pentacene we used the droplet pinned crystallization method. A solution of TIPS-pentacene is made by mixing the solvent m-xylene (sigma Aldrich) and carbon tetrachloride (CCl_4) of Sigma Aldrich with volume ratio 1:1. We have taken a 10 µl solution of TIPS-pentacene with the help of Finn Pipette having different concentration 0.2 $mgml^{-1}$, 0.4 $mgml^{-1}$, 0.6 $mgml^{-1}$, 0.8 $mgml^{-1}$ and 1.0 $mgml^{-1}$, and dropping it onto a silicon substrate ($1cm^2$) with the help of a small piece of silicon wafer (0.4cm x 0.4 cm) called pinner to pin the solution droplet. The optical micrograph of a single crystal having concentration 0.2mg/ml is shown in Fig.1.The silicon substrate is placed on a Teflon slide inside a Petri dish (35mmx10mm). After putting the substrate having grown single crystal inside the Petri dish, it is sealed with para film allowing the solvent to evaporate slowly on a hotplate having temperature 30±1ºC. Aligned crystals are formed within one hour as reported by Hanying Li et al [25]. X-ray diffraction (XRD) measurements for TIPS pentacene powder and single crystal are performed by using a Brucker diffract meter in specular θ/2θ mode. The absorption spectra of TIPS-pentacene solution having concentration 0.2 mg/ml to 1.0 mg/ml are recorded with the help of a Perkin-Elmer UV-Vis spectrometer (Lambda-9). The instrument which is used to measure the electrochemical properties is Cyclic Voltametry (CV) CH1600A with electrochemical work station having platinum disk, plate and a standard calomel electrode (SCE). Saturated calomel electrode is used as a working electrode in a 0.1 mol/l Tetra-butylammonium Hexa-fluorophosphate (Bu_4NPF_6) CH_2Cl_2 solution. The DSC scan of 8.590 mg TIPS-pentacene is performed at 10ºC/min on a Perkin-Elmer DSC 4 in the Nitrogen atmosphere and the scan of CPI-100-AR. The study state photoluminescence is taken on a HITA-CHI 4500 fluorescence spectrometer. FT-IR spectra are obtained in the range from 4000 cm^{-1} to 400 cm^{-1} with a Bruker-Vector 22 infrared spectrometer.

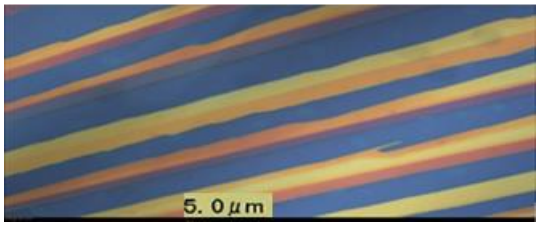

Fig. 1.Optical image of single crystal of TIPS-Pentacene.

Electrical properties of TIPS-pentacene pellet having diameter 20 mm and thickness 0.5 mm is measured on a Novocontrol Alpha high resolution dielectric analyzer (Novocontrol GmbH concept 40, Novocontrol Technology, Germany), equipped with a Novocool Cryogenic system for temperature control. Full detailed related to electrical properties of TIPS-pentacene are described by the author in another article [26].

3. Results and discussions

It is evident from the early studies that small molecules in organic electronics have strong capability to form single crystal which contributes much in carrier mobility. The crystals of conjugated organic molecules have low crystal symmetry structure; they are often monoclinic or triclinic because they possess large symmetry of the molecules [27-30].

The crystals of TIPS-pentacene are triclinic having unit cell parameters a = 7.565Å, α = 89.15º, b= 7.75Å, β = 78.42º, c = 16.865Å and γ = 86.66º. The molecules in TIPS-pentacene are slip-stacked in two dimensions. The XRD pattern of TIPS-pentacene powder and single crystal are shown in Fig. (2) and Fig. (3), respectively. The inter-planar spacing in case of powdered sample is 3.43Å just like in graphite [31, 32]. TIPS pentacene has great anisotropy in its structure; its conductivity strongly depends on the crystalline orientation. The monolayer of single crystals are uniformly distributed over the substrate and the distance between the planes is 16.8Å that corresponds to the long axis of the molecule c = 16.835 Å from the data of the x-ray crystallography. The same values are also reported by the author C. D. Shrew et al and others [33-35]. The crystallinity of the material increases on mixing the solvents. The grain size of the particles increases while the grain boundaries become reduce. As a consequence, the conductivity and mobility increases [36].

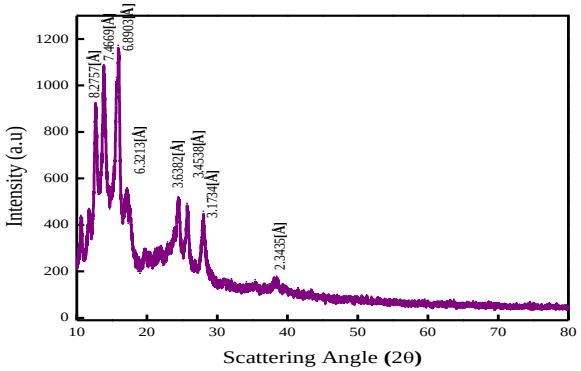

Fig. 2.The XRD of TIPS-pentacene powder

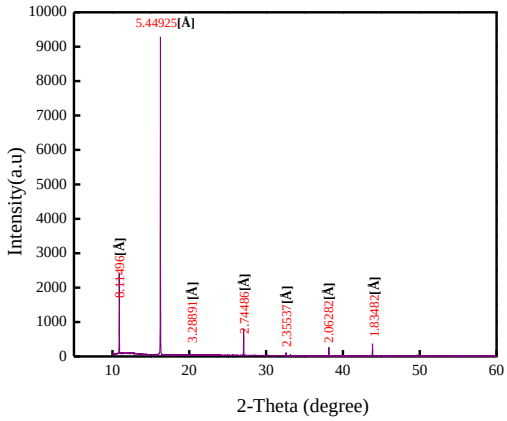

Fig. 3.The XRD pattern of single crystal grown by droplet pinned crystallization method

It is concluded fact that the solvent plays an important role in the formation of a material structure. The X-ray diffraction pattern of TIPS-pentacene powder, single crystal shows that there is a regular arrangement in atoms. Especially, the grown crystal shows an ordered structure. The X-ray diffraction pattern of a single crystal shows that the material is crystalline. A sharp peak at 26.9° due to SiO_2 is observed in powder and single crystal structure. It is clear from the XRD pattern that the crystallinity of the material increases when the material is dissolved in an organic solvent. It means that the solvent plays an important role in crystallinity [36-38].

UV-visible spectroscopy is used to study the molecular energy levels or molecular aggregation effects and it can also be used to characterize the electronic structure [39]. The effects of substituent's on the electronic structure have been studied by measuring their absorption spectra in solutions. Absorption spectra of the TIPS-pentacene are presented in Fig.4. The absorption patterns of the entire derivative are same as the un-substituted pentacene with shift of peaks. The value of optical band gap is 1.8 eV which is the same as cited by Bayram Gunduz et al [40-45].

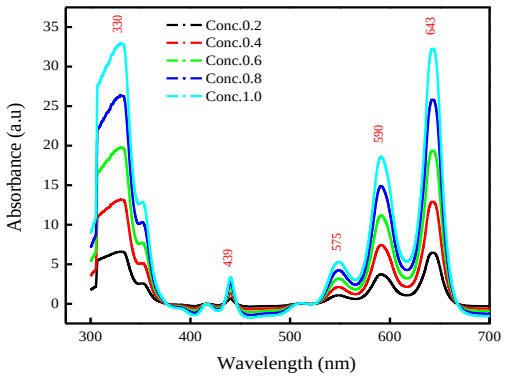

Fig. 4.The UV-spectra of TIPS-pentacene having concentration from 0.2 to 1.0 mg/ml

The presence of a peak at 310 nm, 330 nm indicate the presence of asemiconducting element like silicon in the structure of small molecule.The presence of the peak at 439 nm is due to intermolecular absorption and it also indicates a transition from valence to conduction band, the peaks at the position of 575 nm, 590 nm and 643 nm show that this material is suitable for the application of Optoelectronic devices. The peak at 643nm is due to isotactic point or π-π* transition as reported by David T. James et al [45].The material based on small molecule having no absorption at longer wavelength than 700 nm indicates it is suitable for waveguide configuration [46]. Absorption maxima peak at 643 nm, with few absorption peaks between 400 nm and 600 nm that matches the absorption spectrum reported for TIPS pentacene. The

appearance of the exciton peak around (620 nm) 2 eV is due to the ordered extended polymer structure [47-49].

Cyclic Voltammetry (CV) is used to measure the electrochemical properties of TIPS-Pentacene. It helps in identifying the position of the highest occupied molecular orbital level (HOMO) and the lowest unoccupied molecular orbital level. In organic semiconductor materials the position of these levels helps us in identifying the donor or acceptor material. The onset potential of oxidation (E_{ox}) wave is 0.75 eV while that of reduction (E_{red}) wave is -0.95 eV.

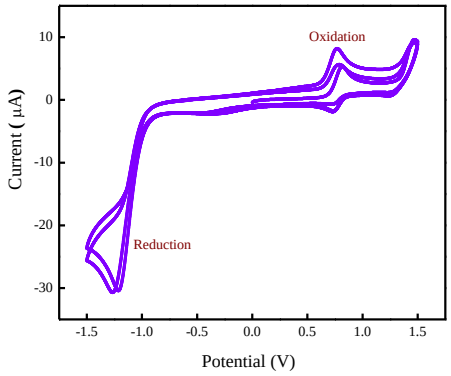

Fig.5. Cyclic Voltammetery (CV) of TIPS-Pentacene

The lowest unoccupied molecular orbital position is calculated by using the relation $E_{LUMO} = -E_{red} + 4.4$ eV, where E_{red} is the reduction potential and the highest occupied molecular orbital position is calculated by the relation $E_{HOMO} = -E_{ox} + 4.4$ eV. The highest occupied molecular orbital (HOMO) of TIPS-Pentacene lies at -5.15eV while the lowest unoccupied molecular orbital (LUMO) lies at -3.45 eV. The electrochemical band gap E_g^{el} value is calculated from the difference between HOMO and LUMO levels and it is found to be 1.70 eV which is in the range of semiconducting materials [47].

In order to study thermal stability, thermal gravimeter of TIPS-pentacene indicates high thermal stability in the range of 50 to 350 ºC. The thermal stability depends on the substituent's group and changes with the change of substituent's positions. Not only the addition of 6, 13-bis (tri-isopropylsilylethynyl) increases the solubility but also the thermal stability. A typical Thermo gravimetric analysis trace is represented in Fig. 6. The inflection at 412 ºC is related to the decomposition process of TIPS-pentacene. In significant decomposition related residue is observed at 498 ºC which is close to the reported value by Jae-Hong Kwon et al [50]. The 60% weight loss is due to breakage of bonds and the curve shows again stability after 600 ºC and this stability confirms the presence of inorganic element in the chain of TIPS-Pentacene. The material melts at about 263 ºC after which its degradation or oxidation takes place as shown in Fig.7. The DSC scan of TIPS-pentacene shows that the material is in crystalline form at low temperature 35 ºC to 63.5 ºC as depicted by the second time heating and cooling curve respectively, a glass transition takes place at 165.51 ºC, that is depicted by the heating curve as shown in Fig. 8[51].

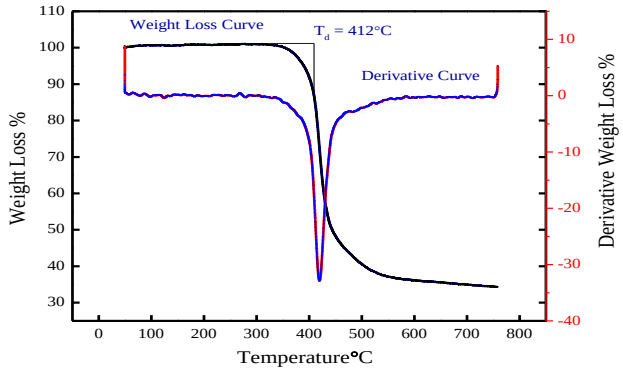

Fig. 6.Weight Loss and Derivative Weight Loss % of TIPS-Pentacene

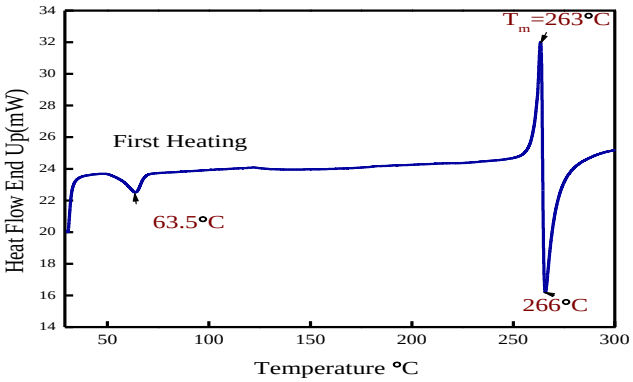

Fig. 7.Differential Scanning calorimeter sketch of TIPS-pentacene

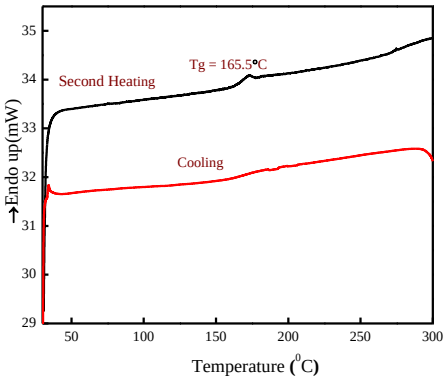

Fig. 8. Differential Scanning Calorimeter Sketch showing the heating and cooling curve

The FTIR spectroscopy of TIPS-pentacene powder is shown in Fig. 9, which reveals the presence of Si-O, Si-C and stretching vibrations of Si-OH at 678 cm^{-1}, 736 cm^{-1} and 888cm^{-1} respectively. The C-Cl stretching peak arises in the range of 590-700 cm^{-1} and C= C stretch is generally present in the range of 1475-1600 cm^{-1}. The bands observed near 1130 cm^{-1} are due to C-C stretching. The bands at 1374 cm^{-1}, 1450 cm^{-1} represent the isolated pentacene anion vibration mode and Si-C stretching vibrations. The bands which are present at the position of 1978 cm^{-1}, 2656 cm^{-1} represents the asymmetric stretching vibrations of methyl(-CH_3) group. The band at the position of 2740 cm^{-1} represents C-H stretching vibration mode and the band at the position 3456 represents the O-H stretching vibration due to moisture content [52-55].

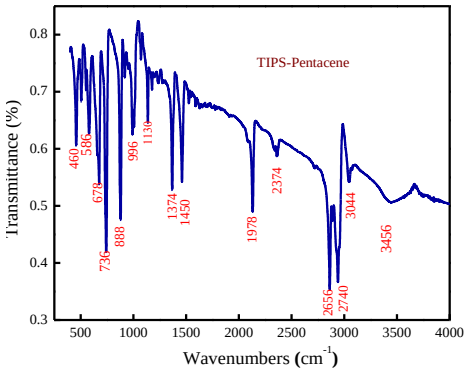

Fig. 9. Fourier transforms infra red spectroscopy of TIPS-pentacene

The photoluminescence spectra of TIPS pentacene solution having different concentrations is shown in Fig. 10. It is clear from the photoluminescence spectrum that the intensity in the blue and red wavelength region is high as compared to other regions of visible spectrum. Maximum photoluminescence is 1.2 x 10^7 at a wavelength of 468 nm. A blue and a red shift is exhibited by the materials at a wavelength of 664 nm, 706 nm having intensity of 9.1x$10^6$and 6.6 x 10^6, respectively. A low photoluminescence intensity of 8.6x10^5 is observed in the green region at a wavelength 560nm. Photoluminescence indicate the presence of infra emission bands as reported by Xiaoxia et al [56]. It is clear from the photoluminescence and UV absorption spectroscopy that the material is sensitive to light because the absorption phenomenon is high in the visible region and two regions red and blue are prominent in absorption [57, 58]. Bathochromic shift and Hypochromic shift are observed in the UV absorption and photoluminescence spectrum.

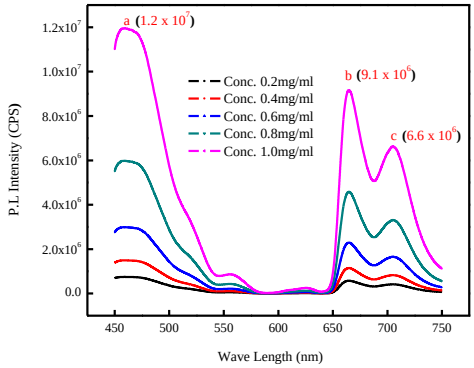

Fig.10. Photoluminescence spectra of TIPS-pentacene powder

Conductivity of the material increases with the increase of temperature and the value of the conductivity lies in the range of a semiconducting material. The maximum value of conductivity is observed at 105 °C and beyond this a decrease occurs as it is depicted in the Fig. 11. This indicates a transition in phase may take place. The initial increase in conductivity with the rise of temperature may be due to the presence of an ordered region. As the material changes phase from ordered to disordered, the conductivity decreases as reported by Hagen Klauk [59].

The log conductivity with the inverse of temperature is shown in Fig. 12. The temperature dependent electronic conductivity obeys Arrhenius rule as the slope is positive up till 105 °C then deviates. It is also reported by Chang-Min Keum that the mobility in TIPS-pentacene degraded after 105 °C due to the change in molecular arrangement or the appearance of a cracks in the material [60].

The temperature dependence conductivity graph shows a deviation behavior from the Arrhenius relation after 105 °C, it may be explained as (i) increase in the lattice vibration, (ii) Phase transition, breakage of bonds and (iv) dielectric breakdown.

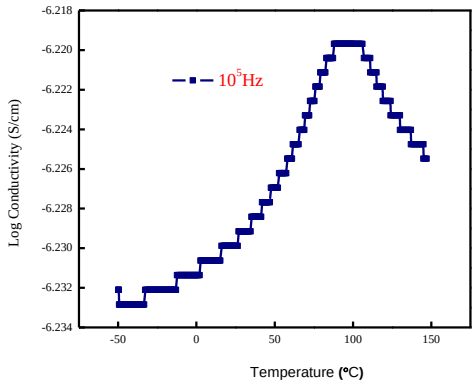

Fig.11. Graph between Log Conductivity (S/cm) versus Temperature (℃)

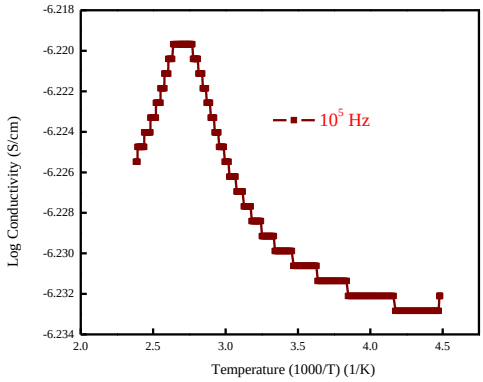

Fig.12. Graph between Log Conductivity (S/cm) versus Temperature (1000/T) (1/K)

4. Conclusions

X-ray diffraction analysis indicates that the material is triclinic having inter-planar spacing of 3.43Å while in case of single crystal it is 16.8Å. UV visible spectroscopy and photoluminescence indicate that the material is photosensitive and it is applicable in optoelectronics. The thermo gravimetric analysis shows that the material decomposes at 412ºC and the differential scanning calorimeter indicates that the material is crystalline at a low temperature. The temperature dependent electronic conductivity obeys Arrhenius rule as the slope is positive up till 105 °C then deviates. It indicates that the material is a semiconductor till 105 °C then a transition in phase takes place. Glass transition takes place at about 165.5 ºC and the melting temperature is 263 ºC. The position of the highest occupied molecular orbital (HOMO) is at -5.15eV and the position of lowest unoccupied molecular orbital (LUMO) lies at -3.45eV. The electrochemical band gap E_g^{el} and optical band gap values are 1.70 eV and 1.8 eV, respectively. These values indicate that the material is semiconducting and applicable to organic field effect transistors, optoelectronics and in electrochemical displays.

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