Schiff base as n-type semiconductor: synthesis, characterization, and diode features

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Abstract
A pyrene-based Schiff base, PyFA, with D-π-A structure was synthesized and the chemical structure was established using IR, NMR, and mass spectrometry. The photoluminescence studies revealed intramolecular charge transfer in PyFA with green fluorescence emission. PyFA with pyrene and fluorene as molecular units was thermally stable up to 300 °C and exhibited an electrochemical band gap of 3.5 eV. The thin films of PyFA exhibited higher carrier concentration, n-type semiconducting property, and a dielectric constant of 6.69. The thin films of PyFA displayed uniform and homogenous surface morphology, which is desirable for device application. A p-n type diode was constructed using PyFA and the diode parameters were extracted from the DC and AC measurements.

Graphical abstract

Keywords Schiff base · n-Type semiconductor · Diode

Introduction
Research is aimed at the efficient design and synthesis of small organic fluorescent molecules because of their potential uses in fluorescent molecular probes, bioimaging, chemosensors, photovoltaics, light-emitting electrochemical cells, and solid-state lighting (Li et al. 2015; Cheng and Zhan 2016; Ding et al. 2016; Gotta et al. 2018; Yang et al. 2018). Conservation and production of clean energy are the most stressed problems...
and one of the ways to address the issue is by designing smart devices that consume less energy (Okon and Biard 2015). The inorganic materials that are currently employed in these electronic devices are crystalline and rigid, need to be highly pure, and demand accurate processing under controlled conditions. Though certain inorganic materials appear promising, they are rare, expensive, difficult to process, and toxic (Zhao et al. 2016). The possible replacement of inorganic with organic semiconductors would lower production costs and enable large-area device development on flexible and lightweight substrates. The precise understanding of structure–property relationships that can be well-utilized to comprehend the characteristics of present devices and predict the best usable material is the key to the conception of high-performance future-generation organic electronics.

The strategic design of optimal highest occupied and lowest unoccupied energy levels in the active layer is an important aspect of an organic electronic device (De Bettencourt-Dias 2007; Binnemans 2009; Eliseeva and Bünzli 2010; Buyruk et al. 2016; Hadsadee et al. 2017). Pyrene is an extensively studied organic fluorophore with strong π-electron delocalization, good thermal features, and ease of functionalization with a wide range of substituent groups (Figueira-Duarte and Müllen 2011), that is capable of both accepting and donating electrons, depending upon the substituents attached (Karuppusamy et al. 2017). The incorporation of a fluorene moiety into the donor and acceptor units has lately gained a lot of interest due to its outstanding characteristics. Fluorene compounds can act as hole transport layer (Shao et al. 2005), electron transport layer (Oldridge et al. 2006; Li et al. 2007), and deep blue-emitting active layer (Gao et al. 2007), and are widely used in OLED applications.

Moreover, though several p-type or hole-transport semiconducting materials have seen a vivid rise in performance over the past decade, significantly less research has been focused on n-type or electron-transporting materials. Hence, in the present study, we report the synthesis of a new Schiff base, PyFA, with fluorene and pyrene moieties, and the structural confirmation of the molecule through spectral techniques. The photophysical, thermal, electrochemical, and electrical features of the molecule have been explored. The n-type semiconducting material could form homogenous and uniform thin films and hence is investigated for fabrication of a diode and the constructed device is further characterized.

### Materials and methods

All chemicals were procured from TCS, Sigma Aldrich, Finar, Spectrochem, and Alfa Aesar. The melting point was determined by the open capillary method and is uncorrected.

Shimadzu FTIR spectrophotometer and 400 MHz Bruker spectrometer were used to record the IR and NMR spectra. The mass spectrum was obtained using Agilent 6430 Triple Quad instrument with electrospray ionization. 1800 Shimadzu and Horiba spectrophotometers were used to record the electronic and emission spectra. The thermogravimetric analysis (TGA) was performed under nitrogen atmosphere using HITACHI thermal analyzer STA7000 series at 10 °C min⁻¹ as the heating rate. The Carl Zeiss EVO 18 analytical scanning microscope (SEM) and INNOVA SPM atomic force microscope (AFM) were used to examine the surface morphological features of thin films. Cyclic voltammetry (CV) measurements were taken at a scan rate of 50 mVs⁻¹ in a three-electrode conventional cell system using a glassy carbon (electroactive area of 0.0771 cm²), Ag/AgCl(KCl sat) and a platinum wire as working, reference and auxiliary electrodes, respectively, using a CH Instrument USA Model 604E series with beta software. All the electrodes were dipped in supporting electrolyte (tetrabutylammonium hexafluorophosphate) solution of 0.1 M in acetonitrile. Keithley Hall Effect set up 3706A was used for conducting the Hall Effect experiments. The computational studies were carried out using B3LYP functions of Schrödinger’s Materials Science Suite with a 6-31G++ basis set in vacuum. The current–voltage (I–V) measurements and impedance studies of the diode were carried out using Keithley 2400 source meter and Hioki LCR meter IM 3635, respectively. PyFA (100 nm), Alq₃ (60 nm), and Al (100 nm) were thermally evaporated (6×10⁻⁶ mbar) using Harshitha vacuum instrument, and PEDOT: PSS was coated using Navson technologies spin coater.

### Results and discussion

#### Synthesis and chemical characterization

**N-(pyren-1-ylmethylene)-9H-fluoren-2-amine (PyFA)**

2-aminofluorene (2 mmol) and 1-pyrenecarboxaldehyde (2 mmol) were dissolved in ethanol and refluxed with a catalytic amount (2–3 drops) of glacial acetic acid at 80 °C for 5 h and the yellow precipitate of PyFA was filtered and washed with ethanol. The synthetic route for PyFA is presented in Scheme 1.

**N-(pyren-1-ylmethylene)-9H-fluoren-2-amine (PyFA)**

Yellow powder (90.14%) m.p: 198–200 °C; FT-IR (ATR, cm⁻¹) (Fig. S1): 3047 (Ar. CH str.), 1689 (C=N str.); ¹H NMR (DMSO, 400 MHz) (Fig. S2): δ 4.031 (s, 2H, CH₂), 7.319–7.356 (t, 1H, 7.6 Hz), 7.401–7.438 (t, 1H, 7.2 Hz), 7.563–7.584 (d, 1H, 8.4 Hz), 7.621–7.640 (d, 1H, 7.6 Hz),
7.798 (s, 1H), 7.942–7.960 (d, 1H, 7.2 Hz), 8.007–8.027 (d, 1H, 8 Hz), 8.142–8.180 (t, 1H, 7.6 Hz), 8.271–8.293 (d, 1H, 8.8 Hz), 8.329–8.351 (d, 1H, 8.8 Hz), 8.390–8.441 (m, 4H), 8.834–8.854 (d, 1H, 8 Hz), 9.333–9.356 (d, 1H, 9.2 Hz), 9.777 (s, 1H, CH=N); 13C NMR (DMSO, 100 MHz) (Fig. S3): δ 37.02, 118.40, 120.45, 121.09, 121.62, 123.62, 124.20, 124.58, 125.61, 125.66, 126.51, 126.79, 127.14, 127.21, 127.35, 127.93, 129.03, 129.42, 129.50, 130.50, 130.64, 131.29, 133.32, 139.94, 141.32, 143.73, 144.83, 151.15, 158.64; MS C30H19N (ESI, m/z) (Fig. S4): 394.15 (M+1).

Photophysical features

Photophysical properties of the active layer in fabricated devices play an important role. Therefore, it is crucial to obtain in-depth knowledge about the electronic transitions in PyFA at the ground and excited states. Two absorption maxima were observed in the electronic spectrum of THF solution of PyFA (1 × 10⁻⁴ M) (Fig. 1A). The absorptions at ~300 and ~400 nm may be due to π − π* and n − π* transitions in the molecule. The optical band gap calculated from the Tauc plot of PyFA (Fig. S5) was found to be 2.3 eV. The solid-state emission spectrum of PyFA (Fig. 1B) exhibited maximum fluorescence at 532 nm emitting green color. The stoke shift calculated for PyFA using Eq. (1) was found to be 13,000 cm⁻¹, and the high value indicated that there is no reabsorption of radiation (Ulla et al. 2014b). The molar extinction coefficient (ε), which is the measure of the strong absorption of light by PyFA calculated using Eq. (2) was found to be 6.60. The high values of log ε suggested that the charge transfer is through π − π transitions (Ulla et al. 2014a).

\[
\text{Stoke shift} = \left(\frac{1}{\lambda_{\text{abs}}} - \frac{1}{\lambda_{\text{em}}}\right) \times 10^7
\]

where λabs and λem are the wavelengths at maximum absorbance and emission, respectively.

\[
\varepsilon = \frac{A}{mC}
\]

where A, m, and C are absorbance, path-length of light, and concentration of PyFA, correspondingly.

The introduction of electron-accepting and donating groups to a molecular system may induce intramolecular charge transfer (ICT), which can alter the photophysical features drastically (Mohan et al. 2020). Therefore, DFT studies were initially performed to create the optimized molecular geometry of PyFA, and further, comprehend the distribution of electron density in the molecular orbitals of PyFA obtained from theoretical studies, and D emission spectra exhibiting the fluorescence response of 1 × 10⁻⁴ M of PyFA with THF/THF-hexane mixtures (λex = 350 nm).
orbital energy levels (Segura et al. 1996). The electronic distribution in HOMO was found all over the molecule, but in LUMO the electron density was localized majorly on the pyrene molecule as observed in Fig. 1C, suggesting the ICT process (Karuppusamy et al. 2017). To confirm ICT process, different hexane fractions were added to $1 \times 10^{-4}$ M solution of PyFA in THF. The emission spectra were recorded with 0–90 volume% of hexane and the variations in fluorescence responses are portrayed in Fig. 1D. The fluorescence emission was found to drastically decrease with increasing hexane content and a hypsochromic shift of 32 nm was observed from 0 to 90% hexane fractions. This interesting ICT process exhibited by the pyrene derivative could be due to the polarization in the molecule leading to the separation of charge creating dipole moment.

**Cyclic voltammetry studies**

Cyclic voltammetry provides qualitative information on the electrochemical properties, and redox potentials of PyFA, which is useful while fabricating electronic devices. The current vs. voltage plot (Fig. 2A) exhibited a quasi-irreversible curve and the redox peak in the positive voltage side in the scanning window range of −3 to 3 V with 100 mVs$^{-1}$ as the scan rate. The oxidation and reduction onset potentials of PyFA were found to be 1.6 and −1.9 eV, respectively. The HOMO and LUMO values were −5.9 and −2.3 eV, with 3.5 eV as the bandgap.

**Thermal characterization**

The ability to withstand higher temperatures without any degradation or deterioration is an important characteristic of a functional material for use in devices, wherein a high current can result in temperature variations (Liu et al. 2018). Hence, the capability of PyFA to withstand operational temperatures was assessed by subjecting it to DSC and TGA measurements. The DSC thermogram as depicted in Fig. 2B revealed a sharp endothermic peak at 200 °C, which corresponds to the melting point of PyFA obtained from the open capillary method. Moreover, the TGA plot of % weight loss vs temperature presented in Fig. 2C displayed two degradation steps: the first one at −320 °C with a weight loss of 29.73% and the second at −640 °C, confirming the thermal stability of PyFA.

**Surface morphology analysis**

Surface uniformity and grain pattern of thin-film layers of functional materials play a vital role while fabricating a device (Ukoba et al. 2018). The morphology of the films formed is significantly connected to the transport properties of the devices. Highly rough surfaces result in a larger interfacial area leading to the growth of conducting regions in the active layer. As a consequence, leakages are observed in the device. The SEM image of thermally evaporated thin films of PyFA as depicted in Fig. 2D shows

![Fig. 2](image-url)

**Fig. 2** A Cyclic voltammogram, B DSC and C TGA thermogram of PyFA. D SEM and E AFM images of a thin film of PyFA
cylindrical-shaped crystalline structures with uniform morphology, which is suggestive of higher conductivity in devices. The AFM image of the thin film of PyFA as presented in Fig. 2E showed uniform surface morphology with a low surface roughness value of 17.4 nm, favorable for device applications.

**Electrical characterization**

For the successful construction of devices, the electronic characteristics of semiconducting materials that act as active layers must be fully understood. The Hall Effect was used to determine the semiconductor type, the charge carrier concentration, and the mobility of electrons and holes. Various quantitative parameters like mobility, resistivity, and charge carrier concentrations of PyFA were determined. The results indicated that the molecule can act as an n-type semiconducting material with a high charge mobility of 2.80 cm²/Vs and carrier concentration of $8.53 \times 10^{13}$ cm⁻³.

Furthermore, the dielectric studies of PyFA were performed from 10 to 5000 Hz. About 100 nm each of PyFA (0.1–0.5 Å/s) and Al (1 Å/s) was thermally evaporated under a vacuum of $6 \times 10^{-6}$ mbar on a pre-patterned ITO plate, which acted as a parallel plate capacitor. The capacitance vs frequency graph as portrayed in Fig. 3A exhibits higher capacitance at a lower frequency. This trend could be attributed to the response of major and minor carriers and interfacial states to the AC. However, at higher frequencies, only the major carriers and some parts of interfacial states respond to the AC, which leads to a sudden decrease in the capacitance value (Chattopadhyay and RayChaudhuri 1993; Lee et al. 2001). Frequency-dependent capacitance of PyFA at 2000 Hz was found to be 0.035 nF.

The dielectric constant is the relative permittivity of a dielectric substance (Patil et al. 2008), which can be calculated using Eq. (3). Frequency vs dielectric constant plot was generated as depicted in Fig. 3B, and the dielectric constant of PyFA at 3000 Hz was 6.69. The higher value of dielectric constant at lower frequency is due to the polarization of PyFA, but the sudden reduction with increasing frequency could be because of the space charge polarization effect (Pontes et al. 2000; Zubko et al. 2006).

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**Fig. 3**  
A Capacitance vs frequency, B dielectric constant vs frequency, C dielectric loss vs frequency and D AC conductivity vs logarithm of angular frequency for PyFA
where $C, d, \varepsilon_0$, and $A$ are capacitance, thickness, permittivity in free space, and active area of the device.

Dielectric loss is the energy absorbed by the medium when AC is passed through it. The loss is zero in an ideal dielectric material. However, in a real dielectric material, some amount of energy is absorbed. The dielectric loss is plotted against frequency as exhibited in Fig. 3C. The low dielectric loss indicated minimal energy absorption by PyFA. The dielectric loss and AC conductivity are interlinked: the AC conductivity increases the eddy current, which in turn increases the dielectric loss. The AC conductivity of the semiconducting material was calculated using Eq. (4). Further, the AC conductivity vs natural logarithm of the angular frequency plot was generated as shown in Fig. 3D. The AC conductivity of PyFA was found to be $\sim 180$ n$\Omega^{-1}$ cm$^{-1}$ at higher frequencies. The decrease in series resistance and the dipole–dipole interactions between the ionic charges might explain the abrupt increase in conductivity with increasing frequency (El-Nemr et al. 2016).

$$\sigma_{AC} = \omega \varepsilon_0 \varepsilon_r \tan \delta$$  \hspace{1cm} (4)

where $\omega$, $\varepsilon_0$, $\varepsilon_r$, and $\tan \delta$ are the angular frequency, permittivity in free space, the dielectric constant of PyFA, and the dielectric loss.

**Device fabrication**

PyFA was used as an n-type of material to construct a p-n diode with device configuration ITO | PEDOT: PSS | PyFA | Alq$_3$ | Al. In this device, spin-coated PEDOT: PSS acted as a hole transport layer on the ITO anode. Thermal evaporated (vacuum of $6 \times 10^{-6}$ mbar) thin films of PyFA (100 nm; 0.1–0.5 Å/s), Alq$_3$ (60 nm; 0.1–0.5 Å/s) and Al (100 nm; 1 Å/s) acted as active layer, electron transport layer and cathode material. The I–V curve was recorded for the diode as depicted in Fig. 4A, which exhibited current in nano-amperes. The log I vs V plot (Fig. 4B) was also generated to calculate the ideality factor (39.7) of the diode. The lower current and high ideality factor observed for PyFA could be attributed to the interfacial defects, which limited the current flow through the device.

Further, the dielectric measurements were performed for the constructed diode, which acted as a parallel plate capacitor. The frequency-dependent capacitance for the device was found to be $\sim 0.6$ nF at higher frequencies from the respective plot as shown in Fig. 4C. Furthermore,
the dielectric constant of the diode was calculated and plotted as exhibited in Fig. 4D, which was found to be 3.35 at 1000 Hz. A decline in the dielectric constant value at higher frequencies might be attributed to the inability of the minor charges and interfacial states to respond to the AC. The minimal dielectric loss was observed in the system as indicated in Fig. 4E. Frequency-dependent AC conductivity was calculated and plotted as depicted in Fig. 4F which was found to be ~28 nΩ⁻¹ cm⁻¹ at higher frequencies.

**Conclusion**

Schiff base PyFA was synthesized and structurally characterized using spectral techniques. The molecule with D-π-A architecture showed an ICT feature, which was established using photophysical studies. Theoretical studies also supported the ICT process in PyFA. TGA thermogram indicated the thermal stability of PyFA, which can withstand temperatures up to 300 °C. The molecule exhibited an electrochemical bandgap of 3.59 eV. The dielectric measurements for PyFA revealed the dielectric constant value of 6.69 of material and AC conductivity of ~180 nΩ⁻¹ cm⁻¹ at higher frequencies. A diode fabricated using PyFA exhibited an ideality factor of 39.7 and a dielectric constant of 3.35 at 1000 Hz. The above results suggest that PyFA can be used as potential n-type material for device applications, and could be further explored as an active layer in OLEDs.

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**Authors' contribution** SK performed methodology and experimental work and was involved in preparing the draft manuscript. DS and DK were involved in conceptualization, supervision, formal analysis, and manuscript editing.

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**Data availability** The datasets generated during and/or analyzed during the current study are available from the first author on reasonable request.

**Declarations**

**Conflict of interest** The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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