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# Design, synthesis, and characterization of TPA-thiophene-based amide or imine functionalized molecule for potential optoelectronic devices

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## Abstract

New sets of molecules containing tri-phenyl-amine (TPA) core and thiophene unit with amide and imine functional groups are designed, synthesized, characterized, and compared. These are solution processable small molecules with high mobility. The newly designed molecules have better solubility due to the C=N (imine) and CONH<sub>2</sub> (amide) moiety as compared to the established molecules with CH=CH (methine) for optoelectronic applications. They have an optimal energy band gap, which indicates their potential utility in a variety of optoelectronic applications. These molecules also show efficient intermolecular charge transfer mechanisms similar to conventional organic semiconducting molecules as evidenced by optical measurements. Density functional theory simulation results show that the localization of the frontier highest occupied molecular orbital is around the TPA core for molecules coupled with imine and amide, and is reasonably stable.

**Keywords:** TPA, Amide, Imine, DFT, Optical properties

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## Background

In the search for inexpensive photovoltaic devices, significant advancements have been made. Particularly, organic dyes and other solution processable small molecules are emerging as promising candidates for photoactive compounds. The advantages of solution processable molecules include high purity, well-defined molecular weight, and easier fabrication steps compared to vacuum deposited methods. Photo-induced charge separation, a key process which occurs when photons have been absorbed by PV material or dye, causes transfer of electrons to nearby accepters. Hence, it can be understood that the donor acceptor (D-A) network inside the system will result in a 'bulk heterojunction' in most photovoltaic devices [1-3]. A D-A network is useful in many cases due to the formation of large interfacial areas, which enhance photoinduced charge separation. One of the promising candidates for such networking is tri-phenyl-amine

(TPA). TPA fulfills most of the above-stated requirements such as strong visible absorption due to D-A structure, solution processability, and an electron rich functional group donor unit [1-3]. In most cases, TPAs and their derivatives possess donor- $\pi$ -conjugated bridge structures [4].

Another class of molecules, thiophene and its derivative, has been proven to be a good candidate for photovoltaic applications because of higher hole mobility due to high intermolecular ordering [5]. It is important to observe that the methine (-CH=CH-) unit, inserted between thiophenes, is widely utilized to extend the photoresponse towards the red region of the spectrum as well as to enhance electron donating ability [4,6]. This methine unit is utilized in polymerization. Some research groups utilized alternative conjugation units for expanding  $\pi$  conjugation and to enhance the stability of dye molecules [7]. It is reported that the inner vinyl group and its high reactivity occasionally causes depletion of absorbance and reduced life time of solar cells [7-9].

In view of these needs, we present two classes of molecules synthesized using TPA [8] and thiophene derivatives. The first category is tris-4-iminophenyl-alkyl-thiophene,

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which contains the imine group. The second group contains tris-4-amidophenyl thiophene.

Tris-4-iminophenyl-alkyl-thiophene is a new solution processable molecule, with TPA as the core and thiophene derivatives as arms bridged through C=N bonding. This class of molecule also has good optoelectronic properties, excellent film forming ability, and solubility in various organic solvents [10]. Some of the applications of imine-based polymers include thin film transistors, light emitting diodes (LED), and other useful devices such as thermostabilizers and high-temperature-resistant composites [11-15]. Some research groups have reported the utility of the imine group in many compounds such as polymer-containing salicylaldimine-based azo ligands and thiophene derivatives [16,17].

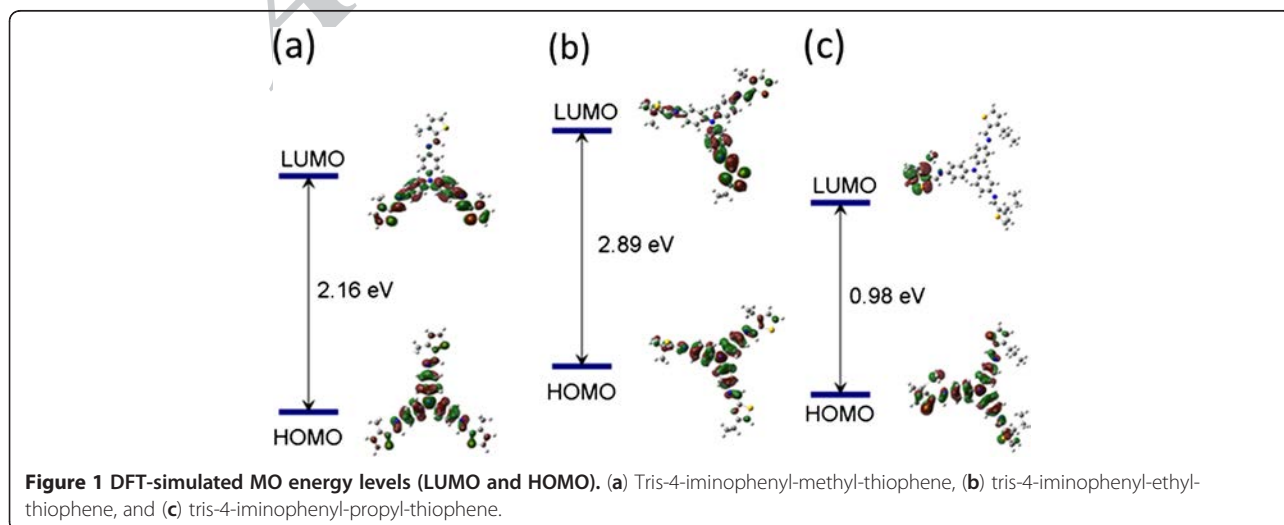
Another molecule which we report is 'tris-4-amidophenyl thiophene'. In this molecule, the TPA core is arm-bridged with thiophene by an amide group. Bushey et al. [18] and Brunsveld et al. [19] synthesized 1,3,5-benzenetrisamides and 2,4,6-trialkoxy-1,3,5-benzenetrisamides, where intermolecular hydrogen bonding is utilized. In many cases, hydrogen bonding is useful for the layering of various discotic materials. Incorporation of amide groups in the side chain causes the attraction of different molecular units, resulting in the formation of columnar assemblies in the ordered layers. Due to the hydrogen bonding, the aggregation behavior of these molecules is different; most of the time, it results in crystalline materials with columnar structure. A goal to attain weak reversible and direct interaction with other neighboring dye molecules can also be achieved, which may be useful to increase the semiconductor film continuity. Another consequence of amide linkage is the observation of red shift in spectra. This red shift is useful for designing the new molecules or polymers with extended chain and a moderate highest occupied molecular orbital-lowest

unoccupied molecular orbital (HOMO-LUMO) gap. A recent study also reveals that conjugate polymer containing amide linkage and thiophene unit has good thermal stability, adequate band gap about 2.21 eV, and strong 2-photon absorption [20]. It is believed that these new solution-processable molecules are excellent candidates for various semiconducting applications due to high mobility and dispersability with other molecules/polymers [21,22].

#### Atomistic simulations

We have conducted density functional theory (DFT) simulations for series of TPA-thiophene coupled molecules to obtain HOMO-LUMO gap information and their geometry prior to experimental synthesis work. To optimize the geometry of molecules, we have used a B3LYP method with triple zeta quality basis set 6-311G(d,p), augmented with polarized *d-functions* for non-hydrogen atoms and polarized *p-functions* for hydrogen atoms. B3LYP is a hybrid exchange correlation functional, which is often used for such calculations. Gaussian 09 code was used for all associated calculations.

Our first set of simulation was for tris-4-iminophenyl-alkyl-thiophene, where we conducted simulations for three different alkyl (methyl, ethyl, and propyl) groups. GaussView-visualized molecular orbitals and their respective energy level diagrams are plotted based on DFT calculations (Figure 1a,b,c). For methyl-modified molecule (tris-4-iminophenyl-methyl-thiophene), HOMO and LUMO are separated with an energy gap of approximately 2.16 eV. The HOMO-1 position is about 1.2 eV below the HOMO level. On the other hand, the LUMO is closely located at about 0.40 eV to LUMO +1. For ethyl group (tris-4-iminophenyl-ethyl-thiophene), the HOMO-LUMO gap is higher at approximately 2.89 eV. The relative position for HOMO-1 is about 1.09 eV below the HOMO



level, while the energy gap between LUMO and LUMO+1 is around 0.10 eV. For tris-4-iminophenyl-propyl-thiophene the, HOMO-LUMO gap is relatively low at around 0.98 eV. It was observed that HOMO-1 is closely located to HOMO at approximately 0.038 eV, while the energy gap between LUMO and LUMO+1 is about 0.91 eV. It is important to notice that for all of three molecules, the density of HOMO is higher at the TPA unit, though it is distributed throughout the molecule. Such distribution is important for the utilization of these molecules as donor units in organic photovoltaic devices [21]. It also can be seen that the density of LUMO is delocalized over the thiophene and imine groups for all three molecules. It is interesting to observe that this delocalization shifted away from the core when moving from the methyl group to the propyl group.

The second set of simulation was for tris-4-amidophenyl thiophene (see Figure 2). This molecule shows a relative high HOMO-LUMO gap of around 3.189 eV. In this molecule, the density of HOMO is higher at the TPA unit compared to the thiophene unit. On the other hand, the density of LUMO is delocalized over the thiophene and amide group, similar to that for tris-4-iminophenyl-alkyl-thiophene. This DFT analysis suggests

that the orbital structures of these molecules closely match with other TPA-thiophene-coupled molecules with a vinyl group. It also suggests that the side chain in the thiophene unit in TPA-thiophene-coupled molecules is important in terms of fine tuning of optoelectronic properties. A relatively low band gap material can be useful for applications such as single-photon sensitivity to telecommunication waves, organic chromophores for near infrared organic light emitting diodes (OLEDs), and other electroluminescence applications [23,24]. For the material with HOMO-LUMO energy gap of 2.16 eV (tris-4-iminophenyl-methyl-thiophene), there is a great potential for application in solution-processable photovoltaic devices with good open circuit voltage [22,25].

### Experimental work

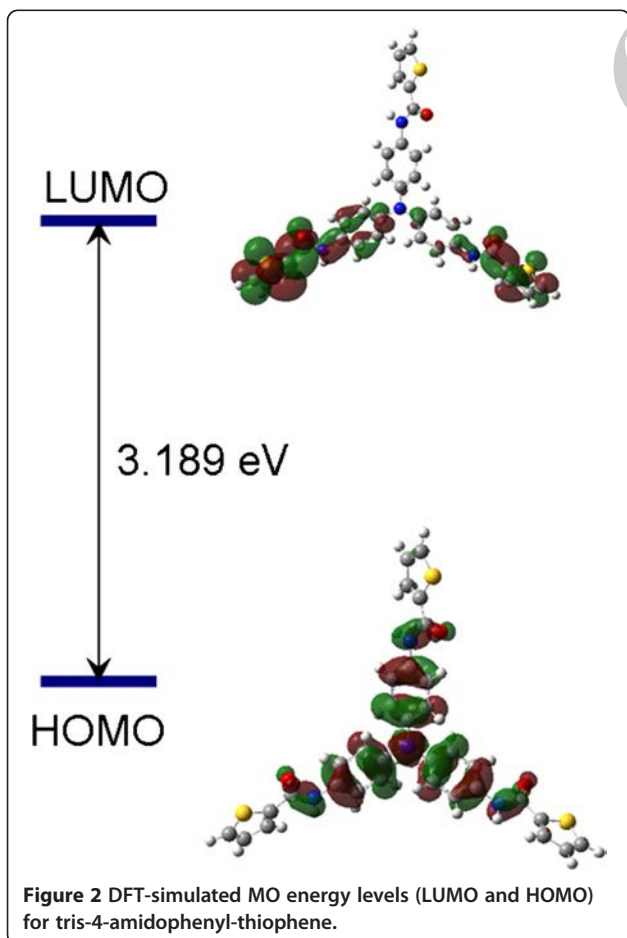
#### Synthesis of tris-4-iminophenyl-methyl-thiophene

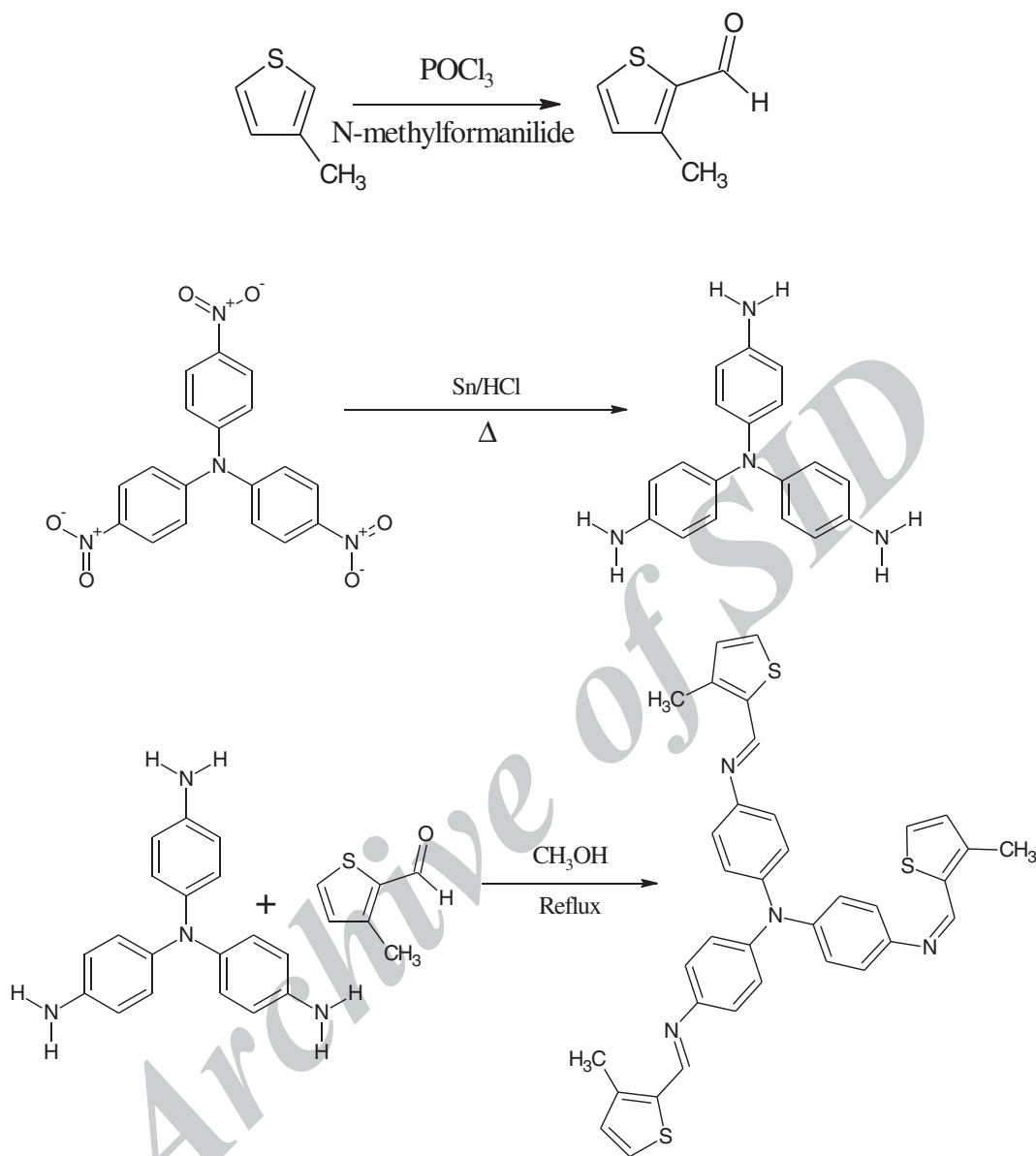
Among tris-4-iminophenyl-alkyl-thiophene group of molecules, we have synthesized tris-4-iminophenyl-methyl-thiophene by the coupling of aminophenylamine with methyl thiophene aldehyde (see Additional file 1: for other details). Synthesis and extraction of methyl thiophene aldehyde were carried out following established procedure [26]. Tris-4-nitrophenyl amine was reduced using Sn/HCl to obtain tris-4-aminophenyl amine. The neutralized cooled filtrate was extracted with dichloromethane. Tris-4-aminophenyl amine was also utilized during tris-4-amidophenyl thiophene synthesis. Methyl thiophene aldehyde and tris-4-aminophenyl amine were used (stoichiometry ratio 3:1) to synthesize tris-4-iminophenyl methyl thiophene. The extracted residue was recrystallized in CH<sub>3</sub>OH, which was dried in vacuum. The tris-4-iminophenyl-alkyl-thiophene is soluble in most common organic solvents and shows excellent film-forming ability. The synthesis steps are shown in Scheme 1.

#### Synthesis of tris-4-amidophenyl thiophene

3-thiophenezoic acid was synthesized by a method similar to that described by Lee et al. [27]. 3-methylthiophene (1 ml) and potassium dichromate (3.6 g) were mixed in distilled water (about 17 mL) and placed in a stainless steel vessel (inner volume approximately 30 mL). The reaction mixture was heated for 12 h at temperature around 200°C. After cooling, the filtrate was acidified to pH 1 to form a precipitate which was collected by filtration and dried to give 0.2 g (30%). The filtrate was extracted with ether (2 × 20 mL).

The newly designed molecule tris-4-amidophenyl thiophene was prepared by a coupling reaction, catalyzed by boric acid [28]. It was achieved by coupling three equivalents of thiophenezoic acid with one equivalent of aminophenylamine (see Additional file 1: for other details). The filtrate was extracted with hexane



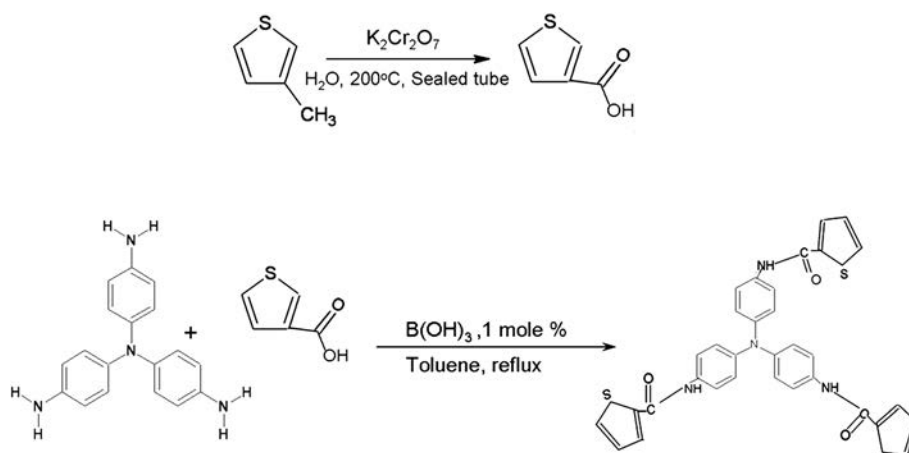


**Scheme 1** Synthesis route of tris-4-iminophenyl methyl thiophene.

after 24 h of reaction. The synthesis steps are shown in Scheme 2. This molecule is gray-white color and shows crystalline behavior. Figure 3 shows a schematic view of the aggregate of tris-4-amidophenyl thiophene molecules, where possible position of hydrogen bonding via side chain amide units is shown. The film-forming ability of this molecule was examined. It was found that the resulting film was rougher than that of tris-4-iminophenyl-alkyl-thiophene (see Figure 4a,b).

#### Absorbance measurements

After detailed and systematic characterization of tris-4-iminophenyl-methyl-thiophene and tris-4-amidophenyl thiophene formation, optical and electrochemical properties were evaluated. An initial assessment of optical properties of these molecules was carried out by absorbance measurements using an Ocean Optics spectrophotometer (Ocean Optics, Inc., Dunedin, USA). Absorbance measurements were carried out in chloroform, ethyl alcohol, and toluene. A background absorbance subtraction



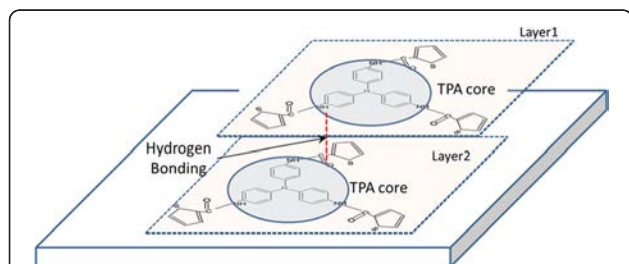
**Scheme 2** Synthesis route of tris-4-amidophenyl thiophene.

(due to each solvent) was applied. Solvents containing these molecule show good absorbance as can be seen in Figures 5 to 6. For tris-4-iminophenyl-methyl-thiophene, two distinct absorbance bands, one in the ultraviolet and the other in the visible region, can be seen (see Figure 5). This observation suggests  $\pi-\pi^*$  electron transitions of the conjugated molecule corresponding to the absorbance bands at around 320 nm and/or 370 nm [29]. An intermolecular charge transfer between the TPA-donating unit and acceptor-end group is also evident from the absorbance band in the visible region (430 to 470 nm) [29]. A peak at about 310 nm is possibly due to the expansion of the  $\pi$ -conjugation system [30]. The band gap of approximately 2.3 eV was calculated based on the onset of absorbance (about 520 nm). These observations suggest that tris-4-iminophenyl-methyl-thiophene should be evaluated for photovoltaic applications. Interestingly, the absorbance behavior significantly matches with other TPA-based molecules [21].

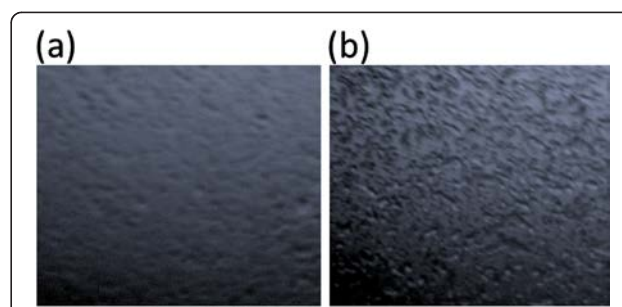
For tris-4-amidophenyl thiophene, two distinct peaks (one at 350 to 550 nm and the other at 750 to 950 nm

were observed (see Figure 6)). The absorbance band (peak centered around 430 nm) in the visible region can be assigned to an intermolecular charge transfer mechanism. A very similar broad absorbance band was also reported for molecules containing TPA with an amide group [7]. Such observation, revealed by strong molar absorptivity, suggests efficient charge transfer between TPA and the side units containing amide and thiophene. The absorbance band at approximately 850 nm is prominent in ethyl alcohol solvent. Such band generally originates from the intermolecular  $\pi-\pi^*$  packing interactions [31]. It is interesting to observe that the band intensity at 850 nm is comparable to that observed at about 430 nm.

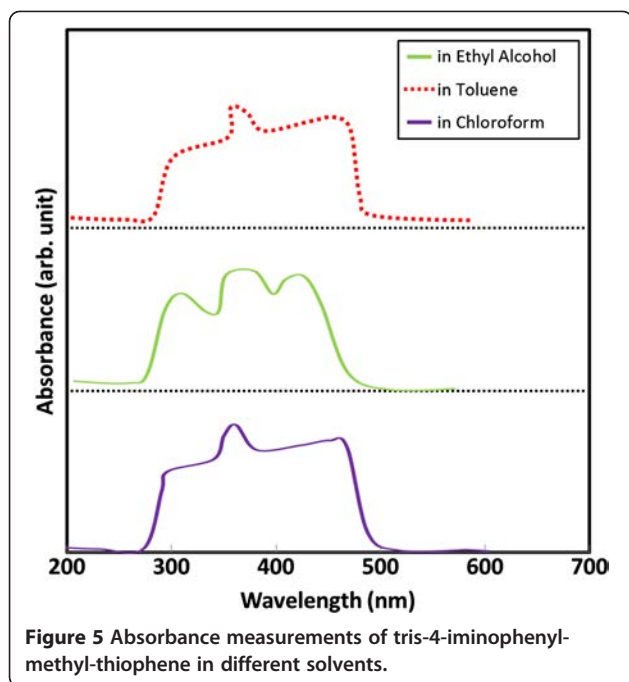
Sometimes, absorption depends on factors such as impinging frequency and size of crystals [32]. However, for our case, these factors were neglected. The difference in absorbance for different solvents can be attributed to possible mechanisms such as intermolecular solute-solvent



**Figure 3** A schematic diagram showing hydrogen bonding between two tris-4-amidophenyl-thiophene molecules.



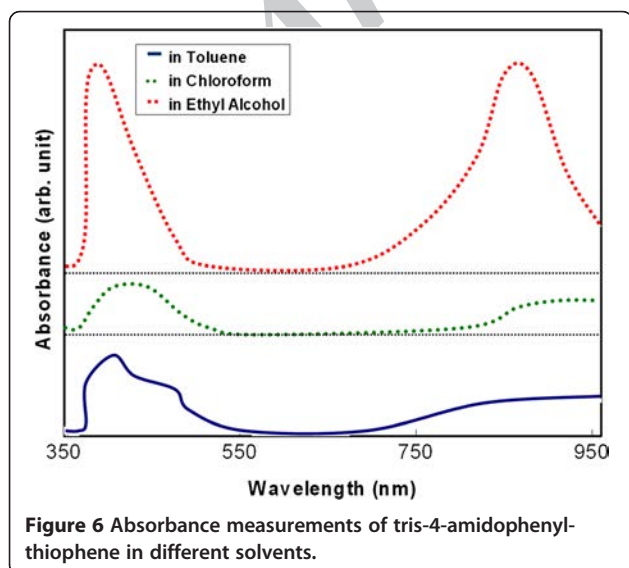
**Figure 4** Optical micrographs (magnification 500x). (a) Tris-4-iminophenyl-methyl-thiophene film surface and (b) tris-4-amidophenyl-thiophene film surface.



interaction, solvent polarity, and hindered electronic transition [33].

#### Electrochemical measurements

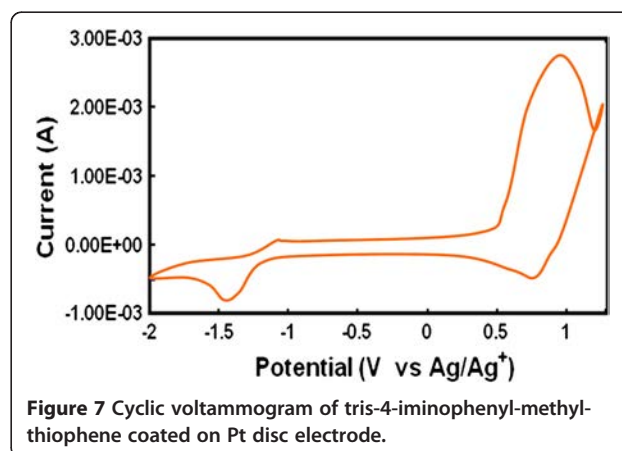
For the estimation of HOMO and LUMO energy levels, cyclic voltammetry (CV) scans were conducted using a Gamry 600 reference potentiostat (Gamry Instruments, Inc., Warminster, USA) equipped with virtual front panel software. An electrochemical cell comprising a platinum disc electrode coated with tris-4-iminophenyl-methyl-thiophene (or tris-4-amidophenyl thiophene),

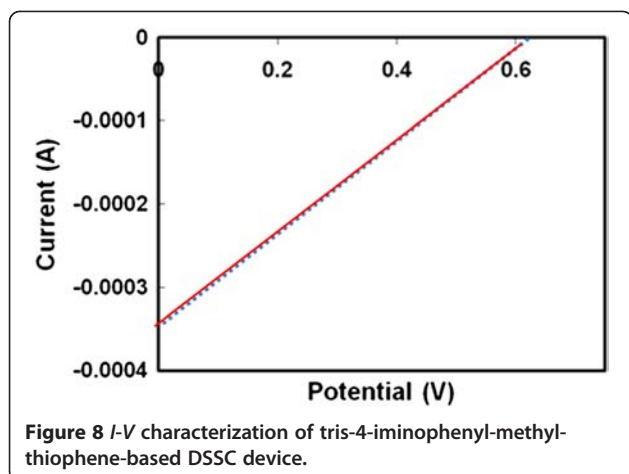


with Pt wire as the counter electrode and Ag/AgCl as the reference electrode, was used for CV scans. Tetrabutylammonium hexafluorophosphate (0.1 mol/L) was dissolved in acetonitrile solution to serve as the redox electrolyte for CV scans. The onset potential data obtained from CV scans are useful for band gap estimations. For tris-4-iminophenyl-methyl-thiophene, the onset oxidation potential is 0.687 V vs. Ag/AgCl, and the onset reduction potential is -1.49 V (see Figure 7). Interestingly, these values closely match with other TPA-based molecules [34]. Using these data, the HOMO-LUMO energy gap of approximately 2.17 eV was obtained for tris-4-iminophenyl-methyl-thiophene [35]. This energy gap is comparable to that obtained from calculated value. Such energy band gap is useful for absorbing a significant portion of the solar spectrum.

#### Photovoltaic device fabrication and characterization

For initial evaluation of photovoltaic performance, a dye-sensitized solar cell (DSSC) was fabricated. A TiO<sub>2</sub> paste was coated on FTO (SnO<sub>2</sub>/F)-coated glass by a glass rod. The sheet resistance of the FTO-coated glass was about 17 Ω/square. After sufficient drying, TiO<sub>2</sub>-coated electrodes (thickness about 40 μm) were annealed at 400°C for 1 h. The sensitization of TiO<sub>2</sub>-coated substrate was carried out using ethanol solution of tris-4-iminophenyl-methyl-thiophene for a short duration of approximately 30 min. A KI (0.1 M)/I<sub>2</sub> (0.05 M) redox electrolyte was used as charge-compensating solvent. Another FTO-coated substrate of similar size was coated with graphite as the counter electrode. Electrical performance (see Figure 8) of DSSC device was measured during incident illumination of 0.25 mW/cm<sup>2</sup>. A device with an active area of around 1 cm<sup>2</sup> shows an open circuit voltage of about 0.62 V and a short circuit current of approximately 0.35 mA. Although novel symmetric dye structures have not been much utilized for DSSC due to inefficient directionally charge transfer and anchoring





capabilities, however, there is scope of improvement by enhancing the charge transfer rate at the organic–inorganic junction and/or increasing the duration of TiO<sub>2</sub>-coated substrate sensitization.

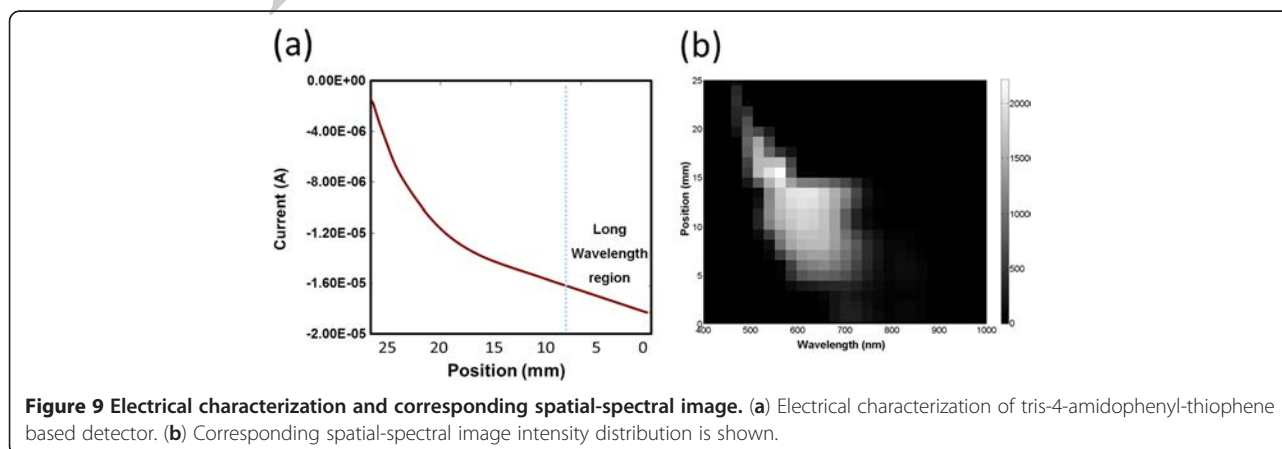
#### Detector fabrications and characterizations

For the evaluation of detecting capabilities of tris-4-amidophenyl thiophene, a device was fabricated. An ethanol solution of tris-4-amidophenyl thiophene was used to make a thin layer. An FTO-coated glass substrate was used for coating. For light dispersion, prism-lens combination was used. A fiber optics illuminator was used as the light source. An incident illumination was about  $3.2 \times 10^4$  lx at wavelength of around 550 nm. Electrical characterization (see Figure 9a) of a device was conducted using a Gamry reference panel equipped with the Virtual Front Panel Software (Gamry Instruments, Inc.). The device was mounted on a linear micrometer stage, placed in the plane where there is maximum lateral separation of

the dispersed wavelengths. It can be seen that the current response of the device is higher at long visible and IR wavelength regime (wavelength greater than 650 nm). The spatial-spectral distributions in the image plane are shown in Figure 9b.

#### Conclusions

We have designed and synthesized new sets of molecules containing TPA and thiophene coupled by imine or amide group. These small semiconducting molecules are useful because of performance, reproducibility in synthesis, ability to functionalize, and ease of purification steps. Tris-4-iminophenyl methyl thiophene absorbs a significant portion of the visible spectrum, which suggests its potential utility for photovoltaic applications. On the other hand, tris-4-amidophenyl thiophene shows two distinct absorbance peaks including one in longer wavelength region in the absorbance spectra, suggesting strong  $\pi$ - $\pi^*$  packing interactions. Using DFT simulations, the HOMO-LUMO gap of tris-4-iminophenyl methyl thiophene molecule was obtained at around 2.17 eV, which closely matches with experimental results. This energy band gap is within optimal band gap range useful for long wavelength absorption. DFT simulations also show that the band gap of TPA-imine-thiophene-coupled molecules can be tuned by changing side alkyl units attached to thiophene. Photovoltaic activity of tris-4-iminophenyl methyl thiophene is confirmed by dye-sensitized solar cell response; however, the improvement in the device performance is needed. Tris-4-amidophenyl thiophene shows enhanced current response for long wavelengths of about 700- to 800-nm illumination. Other experiments related to the synthesis and utility of tris-4-iminophenyl-ethyl-thiophene, tris-4-iminophenyl-propyl-thiophene, and tris-4-amidophenyl thiophene are in progress.



## Additional file

**Additional file 1: DFT simulation, Raman spectroscopy, FTIR characterization, and NMR and mass spectroscopy.**

### Competing interest

The authors declare that they have no competing interests.

### Authors' contribution

PKS carried out the experiments and characterizations, analyzed the data, conceptualized the research, and drafted the manuscript. AS designed the molecules and synthesis schemes, and systematically interpreted the characterizations. YZ conducted the density functional simulations and written a portion of the manuscript. MLF provided guidance at various stages of study and reviewed the manuscript. All authors read and approved the final manuscript.

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