Organic Semiconductors

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Crystal Engineering of Dual Channel p/n Organic Semiconductors by Complementary Hydrogen Bonding**

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Abstract: *The supramolecular arrangement of organic semiconductors in the solid state is as critical for their device properties as the molecular structure, but is much more difficult to control. To enable supramolecular design of semiconducting materials, we introduced dipyrrolopyridine as a new donor semiconductor capable of complementary hydrogen bonding with naphthalenediimide acceptors. Through a combination of solution, crystallographic, and device studies, we show that the self-assembly driven by H bonding a) modulates the charge-transfer interactions between the donor and acceptor, b) allows for precise control over the solid-state packing, and c) leads to a combination of the charge-transport properties of the individual components. The predictive power of this approach was demonstrated in the synthesis of three new coassembled materials which show both hole and electron transport in single-crystal field-effect transistors. These studies provide a foundation for advanced solid-state engineering in organic electronics, capitalizing on the complementary H bonding.*

Organic π-conjugated materials are attractive semiconductor components for a number of optoelectronic applications, particularly because of their tunable properties which are accessible through synthetic chemistry. The discrete molecular characteristic of organic semiconductors (OSCs) presents both a challenge, in controlling solid-state assembly through weak intermolecular forces, and an opportunity for supramolecular control of electrical constituents at the nanoscale. A number of different interactions have thus been used for self-assembly of π-conjugated materials, including H bonding, ionic, π-π, and van der Waals interactions.[1] Controlling the supramolecular organization of OSCs is of paramount importance for devices such as organic field-effect transistors (OFET),[2] light-emitting transistors[3] and photovoltaics (OPV),[4] where charge generation and transport is dictated by solid-state packing (π-π interactions) and nanoscale morphology. Progress toward the optimal packing motifs has largely been empirical, with high charge mobility compounds tending toward planar, fused π-conjugated structures.[5] Limited success toward rational molecular-level control of solid-state structure in single-component OSC devices (e.g., OFET) has been demonstrated using, for example H bonding,[6] sterically bulky substituents,[5b] and van der Waals interactions of alkyl substituents.[7] Controlling the solid-state structure of bicomponent OSC devices (e.g., OPV) is much more difficult and currently not practical. Thus, achieving the optimal phase separation between donor and acceptor in p-/n-bulk heterojunction OPVs[8] rests primarily on empirical formulation of the OSC layer using additives[9] and various annealing conditions.[10]

Assembly of electronic materials by H bonding has been explored since the time of organic metals,[11] and more recently in the construction of H-bonded ferroelectrics.[12] In OSCs, the key materials of today's organic electronics, H bonding have received a surprisingly limited, although quickly growing attention. Efficient charge transport in H-bonded materials has been realized in OFET devices[13] and several studies have connected self-complementary H bonding to enhanced π···π interactions in the solid state.[13b,d] A potentially powerful, yet practically unexplored approach[14] is directed coassembly of bicomponent OSCs through complementary H bonding. Ambipolar transport in OFETs was demonstrated for the H-bonded perylenediimide/oligophenylene-vinylene pair, but the "soft" (amorphous) nature of this material led to very low charge mobilities (10^{-7} $cm^2 Vs^{-1}$), and more importantly, precludes structural characterization of the heterojunction.[14b]

Here we present a crystal-engineering approach to control the morphology in bicomponent semiconductors. We use diphenyldipyrollopyridine DP-P2P[15] as a new p-type semiconductor with an integrated H-bonding moiety complementary to naphthalenetetracarboxydiimides (NDIs) and related n-type semiconductors (Figure 1 a). We show that complementary H bonding between π-conjugated OSCs enhance π-π interactions in the solid state and enable fine-tuning of the p-/n-heterostructure. Furthermore, the coassembly of DP-P2P and NDI components affords molecularly defined hole/electron channels which maintain the charge-transport properties of the individual donor/acceptor components.

NDI and related rylenetetracarboxydiimides are among the most promising n-type organic semiconductors, the properties of which can be easily tuned through *N*-[16] and/or π-substitution.[17] In OFETs, they show record-high electron mobilities;[18] as acceptor components in OPV they are only second to fullerene derivatives.[19] When stripped of the usually employed alkyl substituent, the cyclic imide group of NDI presents a three-point H-bonding functionality with hydrogen acceptor–donor–acceptor (A-D-A) structure, thus allowing for high-fidelity coassembly with donor–acceptor–donor (D-A-D) complementaries.

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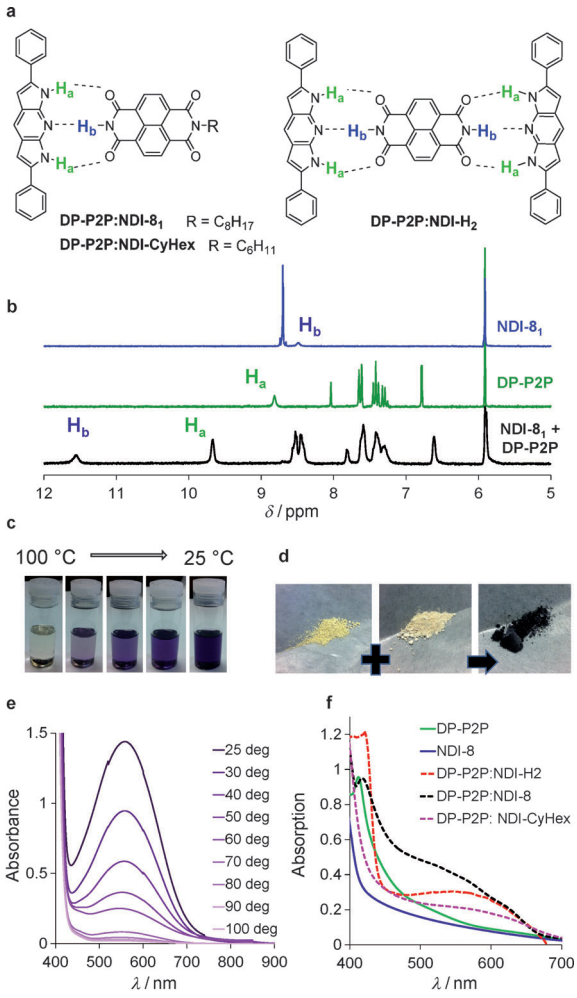

Figure 1. a) H-bonded coassemblies of DP-P2P with NDI. b) ^{1}H NMR spectra of DP-P2P, NDI-8$_1$, and their coassembly in [D$_2$]TCE (70 °C, 8.8 mM). c) Thermochromism of DP-P2P/NDI-8$_1$ solution in TCE (2.0 mM). d) DP-P2P and NDI-8$_1$ powders and their coassembly formed by coevaporation. e) Variable temperature UV/Vis spectra of DP-P2P:NDI-8$_1$ in TCE. f) UV/Vis spectra of thin films of DP-P2P:NDI-8$_1$ prepared by coevaporation.

The electron donor–acceptor properties of the designed semiconductors were studied by cyclic voltammetry (see the Supporting Information). Assuming the ferrocene/ferrocenium couple at −4.8 eV versus vacuum,[20] the HOMO of −5.1 eV and LUMO of −3.8 eV were determined for DP-P2P and monoalkyl-NDI, respectively. These energies are in line with DFT calculations (B3LYP/6-31G(d): DP-P2P HOMO = −5.10 eV, NDI-CH$_3$ LUMO = −3.48 eV) and are appropriate for ambipolar charge transport.

The complementary position of H-bond donor and acceptor centers (DAD···ADA) in P2P and NDI leads to strong binding which is worth 19.6 kcal mol^{-1} in the gas phase (according to the DFT calculations, see the Supporting Information). This is stronger than the interaction of NDI with 2,6-diaminopyridine (17.2 kcal mol^{-1}) and the A···T nucleobase pairing (16.2 kcal mol^{-1}). Strong H bonding is manifested by downfield shift of the NH protons (about 3 ppm for NDI-8$_1$, 1 ppm for DP-P2P) when both compounds are mixed in [D$_2$]tetrachloroethane ([D$_2$]TCE) solution (Figure 1b). While limited solubility precluded concentration-dependent NMR studies, the coassembly can be followed by variable temperature ^{1}H NMR spectroscopy which shows dissociation of the complex at elevated temperature (see the Supporting Information). Interestingly, these solutions display a thermochromic behavior, where a hot (>100 °C) colorless solution of both components leads to a reversible purple coloration upon cooling, which increases in intensity until eventual precipitation of the complex (Figure 1c). The stoichiometric ratio of the precipitated complex was confirmed by ^{1}H NMR spectroscopy. Although both individual components are fluorescent (in solution and in the solid state), their interaction leads to complete quenching of fluorescence, which suggests fast electron transfer between the donor and the acceptor.

The appearance of the bathochromically shifted absorption (λ_{max} = 558 nm in TCE, 543 nm in dichlorobenzene) is indicative of the ground-state charge-transfer (CT) interactions between the donor and acceptor components (Figure 1e). This was not expected considering relatively weak electron donor/acceptor abilities of the components and their separation in the H-bonded complex. No CT band was observed when DP-P2P is mixed in solution with non-H-bonding acceptors: either dialkylated NDI-8$_2$, or the monoanhydride analog of NDI-8$_1$ (a stronger electron acceptor). The absence of a CT band in high polarity solvents such as DMSO (which commonly favors CT complexation) further supports the key role of H bonding in enforcing the charge transfer from DP-P2P to NDI.

The CT band likely arises from π-stacking of H-bonded complexes, where the increased π-surface enhances the aggregation through π-π interactions. Indeed, time-dependent DFT calculations (M062X/6-31G(d)) of the gas-phase optimized DP-P2P:NDI-CH$_3$ complex predicts no absorption above 400 nm; the π-electron systems of the donor and acceptor are separated by H bonds and the corresponding charge-transfer (HOMO–LUMO) transitions have zero oscillator strength (f). On the other hand, the π-stacked dimers of DP-P2P:NDI-CH$_3$ complex are predicted to have a moderate CT transitions (DP-P2P→NDI-CH$_3$ λ_{max} = 540 nm, f = 0.017 and 493 nm, f = 0.059, for co- and anti-parallel dimers, respectively) closely resembling the experimental observations (see the Supporting Information). Thus, H bonding enforce the D-A interactions, creating CT states which are absent in donor–acceptor pairs without H-bonding interactions.

The same DP-P2P:NDI heteroassemblies could also be prepared by coevaporation of a grinded mixture of the components in vacuum. Powder XRD (see the Supporting Information) shows identical diffraction pattern for solution and vapor-grown cocrystals and confirms absence of crystalline phases of individual components. UV/Vis spectra of films grown by sublimation show similar low-energy CT bands extending past 600 nm (optical band-gaps of about 1.8 eV, Figure 1f).

X-ray crystallographic analysis of vapor-grown DP-P2P:NDI-8$_1$ shows a segregated stack structure, with alter-

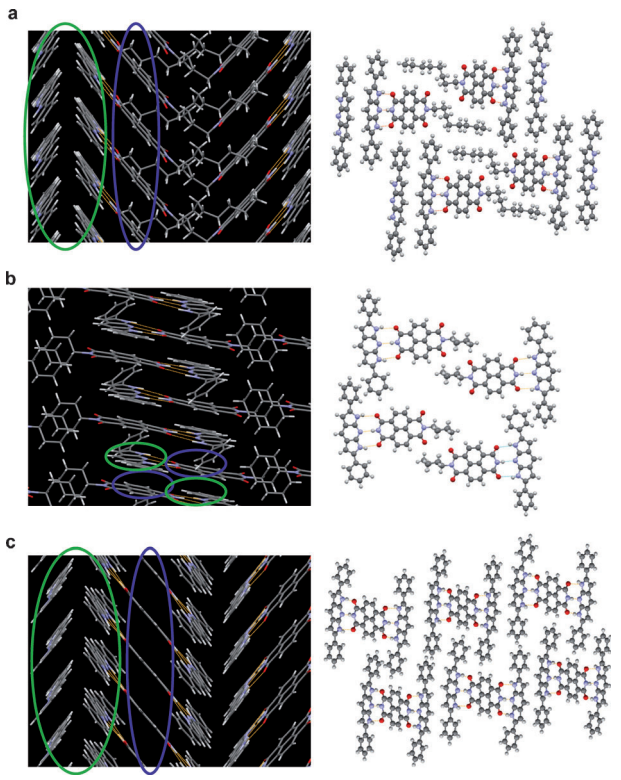

Figure 2. Polarized optical images (top, 1 mm × 1 mm) and X-ray single-crystal analysis (middle/bottom) of a) DP-P2P:NDI-8_1, b) DP-P2P:NDI-CyHex, and c) DP-P2P:NDI-H_2. p-type DP-P2P channels (green) and n-type NDI channels (blue) are highlighted.

nating dimerized (DD···AA···DD···AA) columns (Figure 2a). The D…A boundary, which defines the p-n junction, is controlled by three-point H bonding between DP-P2P and NDI-8_1 molecules. The short N···N (2.788(7) Å) and N···O (2.915(6 Å) distances are indicative of strong H bonding. The molecules in the donor stacks align into a herringbone motif, where DP-P2P in adjacent columns are self-interacting in an edge-to-face orientation, with a herringbone angle (the angle between the molecular planes) of 70° (cf. 53° for pentacene and 64° for isoelectronic diphenylbenzodithiophene).[5b] Such arrangement creates a quasi two-dimensional hole transport pathway, which is most commonly found in high-mobility organic semiconductors. A similar herringbone packing motif is also found in pure DP-P2P single crystals (see the Supporting Information).[21] In the acceptor stacks of DP-P2P:NDI-8_1, the columns of co-parallel NDI molecules are separated by alkyl chains, forming isolated one-dimensional transport pathways with π···π distance of about 3.4 Å and 42° angle of slippage (along the long molecular axis). This slippage also allows for a partial donor–acceptor π-overlap (short C···C contacts [3.176(7) Å]), explaining the observed CT absorption.

The predictable nature of P2P···NDI H bonding allows fine-tuning of the heterostructure by small structural changes to the individual components. This was readily achieved by varying the *N*-substituent on the NDI. Thus, introducing a bulkier secondary alkyl (cyclohexyl, CyHex) chain disfavors slipped π-stacking arrangement of NDI moieties found in DP-P2P:NDI-8_1.[22] Consequently, the DP-P2P:NDI-CyHex co-crystals show a mixed stack arrangement where the DP-P2P molecules sit directly above NDI (Figure 2b). This enhances the donor–acceptor π-interactions but disrupts the continuity of the donor and acceptors domains.

Using unsubstituted NDI-H_2 with two H-bonding imide groups predictably leads to a 2:1 donor/acceptor stoichiometry in DP-P2P:NDI-H_2. The other details of packing are highly reminiscent of DP-P2P:NDI-8_1; the molecules form segregated donor/acceptor channels composed of herringbone-packed donor and slipped π-stacked acceptor, with partial donor–acceptor π-overlap (Figure 2c). Together these results demonstrate the ability to tune the packing characteristics by ensuring the fixed stoichiometry and the geometry of the D···A boundary by H bonding and then tweaking the molecular size/shape of the constituents.

Single-crystal transistors were prepared for the individual components (Figure 3a) and compared to single cocrystal devices. DP-P2P revealed p-channel FET behavior with hole mobility of 0.027 $cm^2 V s^{-1}$ and relatively low threshold

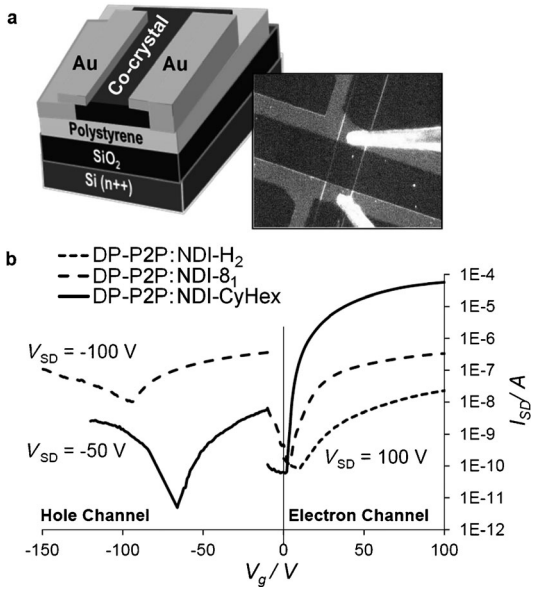

Figure 3. a) Device structure and optical image of single-crystal OFETs. b) n-type and p-type channel transfer characteristics of donor–acceptor cocrystal devices (V_g = gate voltage, V_{SD} = source-drain voltage, and I_{SD} = source-drain current).

voltage of −15 V (see the Supporting Information). The electron mobility of NDI-CyHex ($\mu_e = 0.17$ $cm^2 V s^{-1}$) was slightly higher than that of NDI-8_1 ($\mu_e = 0.081$ $cm^2 V s^{-1}$). These values are within the range typically observed for dialkylated NDIs, demonstrating the ability for efficient electron transport in the presence of the acidic imide proton.

All three cocrystals displayed electron mobility with threshold voltages below 10 V (Figure 3b and the Supporting Information). While DP-P2P:NDI-H_2 was found to be unipolar ($\mu_e = 0.00034$ $cm^2 V s^{-1}$), FET devices using DP-P2P:NDI-8_1 and DP-P2P:NDI-CyHex also exhibited hole transport. DP-P2P:NDI-8_1 showed relatively balanced ambi-

polar transport with hole and electron mobilities of 0.043 and 0.089 $cm^2 V s^{-1}$, respectively. These charge mobilities are among the highest reported for cocrystals,[23] and are more than five orders of magnitude higher than the previously studied perylenediimide/oligo(phenylenevinylene) H-bonded coassembly.[14b]

Interestingly, despite the absence of pure donor/acceptor channels in the DP-P2P:NDI-CyHex crystal, ambipolar transport with high electron mobility ($\mu_e = 0.22\ cm^2 V s^{-1}$) was still maintained, which likely occurs by superexchange mechanisms.[24] The lower hole mobility in DP-P2P:NDI-CyHex ($\mu_h = 0.00041\ cm^2 V s^{-1}$) may be related to the distortion of the DP-P2P (38.5° twist of the phenyl ring) observed in this crystal structure.

In conclusion, we have demonstrated effective crystal engineering of p-/n-type dual channel organic semiconductors. Taking advantage of the complementary A-D-A···D-A-D H bonding between the electron donor (dipyrrolopyridine) and acceptor (NDI) we show rational control of the stoichiometry and packing characteristics in these ambipolar cocrystals. Altering the substituents on the NDI nitrogen controls the supramolecular arrangement, into either segregated or mixed stacks of the donor and acceptor components. Unexpectedly, we found that H bonding enforces ground-state charge-transfer interactions manifested in new long-wavelength absorption (not observed in these weak donor–acceptor pairs without H bonding). Field-effect transistor studies show that highly acidic NH protons do not adversely affect either hole or electron transport in the crystalline state. These findings reveal that the donor/acceptor heterojunction morphology can be controlled through predictable complementary H bonding without disrupting charge-transport properties of the individual components. The predictive power of H-bonding supramolecular design differentiates this work from the recent studies on semiconducting cocrystals[23,24b] and paves a way for structural control of donor/acceptor blends in a variety of organic electronic devices, including solar cells.[25,26]

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