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Near-IR absorption and photocurrent generation using a first-of-its-kind boron difluoride formazanate non-fullerene acceptor†

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Herein, we report the synthesis and characterization of the first non-fullerene acceptor (NFA) containing a boron difluoride formazanate (BF2fz) core end-capped with N-annulated perylene diimides (PDIs). Electronic coupling between the BF₂fz core and the PDI endcaps enabled tuning of the lowest unoccupied molecular orbital, leading to near-panchromatic optical absorption. Post-deposition solvent vapor annealing of the new NFA resulted in a significant red-shift in the optical spectra, which stretched into the near-IR. Proof-of-concept organic photovoltaic (OPV) devices were constructed to demonstrate the potential of this new material as an NFA. SVA treatment of the active layer resulted in a 2-fold increase in power conversion efficiency (PCE), due mainly to increases in the BF,PDI2 generated photocurrent that extended into the near-IR.

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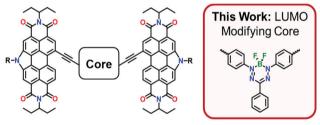
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Introduction

For applications in organic photovoltaics (OPVs), π -conjugated materials are perfectly suited for use as non-fullerene acceptors (NFAs) because both physical and optoelectronic properties may be optimized to match the electron-donor material with which they are paired in the active layer. 1,2 One class of nonfullerene acceptors (NFAs) recognized for high redox and thermal stability, as well as high molar absorptivity, are perylene diimides (PDIs).^{3,4} Despite such favorable qualities, the efficiency of charge-extraction for PDI-based NFA materials is often hindered by the formation of large π - π stacking domains throughout the BHJ. To promote more favorable phase separation in the BHJ, PDI self-assembly may be disrupted through either direct dimerization of PDIs⁵⁻⁸ or insertion of a core between the PDI-moieties.⁹⁻²⁰

The Welch research group has previously explored the optoelectronic influence of thienoisoindigo, thienyl- and pyridyl-diketopyrrolopyrrole cores that were acetylene-linked to N-annulated PDI (Fig. 1).²¹ Strong electronic communication between the cores and PDI endcaps adjusted the highest occupied molecular orbital (HOMO) energy level of these molecules, affording nearpanchromatic absorptions ideally suited for OPV. In search of a core that could achieve similar absorptivity, while instead



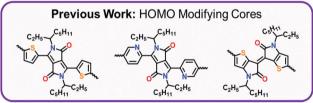


Fig. 1 Depicting previously investigated HOMO-modifying and newly synthesized LUMO-modifying N-annulated PDI-core-PDI materials. R = hexyl (HOMO modifying cores) or 2-ethylhexyl (LUMO modifying core).

influencing the lowest unoccupied molecular (LUMO) energy level, we targeted an acetylene-terminated boron difluoride formazanate (BF₂fz), a chromophore popularized by the Gilroy research group. 22-24 The properties of BF2fz materials are highly sensitive to structural variation at the N-aryl substituents, 25-27 meaning the optoelectronic properties can be tuned by the choice of endcap material. In this contribution, we report the synthesis, characterization, and NFA capability of a new π -conjugated material comprised of a BF2fz core acetylene-linked to N-annulated PDI endcaps (BF₂PDI₂).

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Characterization

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The BF₂fz core²² and N-annulated PDI (Br-PDIN-EH)⁸ end caps were connected via a Sonogashira cross-coupling reaction to generate BF₂PDI₂ (see ESI,† for full experimental details). The identity of **BF₂PDI₂** was confirmed by ¹H, ¹¹B, ¹³C, and ¹⁹F NMR spectroscopy, as well as MALDI TOF mass spectrometry and CHN elemental analysis (Fig. S1-S7, ESI†). The ¹¹B and ¹⁹F NMR spectra exhibited diagnostic 1:2:1 triplet and 1:1:1:1 quartet coupling patterns, respectively.²⁵ Moreover, the ¹H NMR spectrum showed no evidence of a formazan related N-H peak (at ~ 15 ppm), ²² strongly suggesting the BF₂fz core remained intact throughout synthesis and purification.

The thermal properties of BF₂PDI₂ were measured using a combination of differential scanning calorimetry and thermal gravimetric analysis (Fig. S8 and S9, ESI†). No obvious glass transitions or melting points were observed between 100-300 °C, while the onset of molecular decomposition was not observed until > 300 °C. The high thermal stability observed in $\mathbf{BF_2PDI_2}$ may be attributed to significant π -conjugation between the BF₂fz core and the N-annulated PDI endcaps. This notion was supported by the density functional theory (DFT) optimized structure of BF₂PDI₂ at the B3LYP-6-31G(d,p) level of theory (Fig. S14, ESI†). The PDI units were nearly co-planar with each other, with only a small degree of bending caused by the BF2fz core. This DFT optimized 'dragonfly' structure corresponded well with previously determined X-ray crystallography structures for compounds using the same BF₂fz core.^{23,24}

Next, the electronic properties of BF₂PDI₂ were probed using cyclic voltammetry (CV), with additional information provided by DFT calculations. The CV of BF2PDI2 was comprised of three irreversible oxidation events, as well as four fully reversible reduction events (Fig. 2). While the number oxidation events could be determined by differential pulse voltammetry (Fig. S13, ESI†), the precise electrochemical origin may not be readily

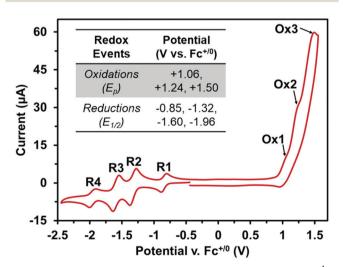


Fig. 2 Cyclic voltammogram of BF_2PDI_2 , measured at 100 mV s⁻¹ in CH_2Cl_2 under argon with 0.1 M TBAPF₆ supporting electrolyte (WE = glassy carbon, CE = Pt-wire, pseudo-RE = Ag/AgCl). Inset are all measured oxidation (Ox) and reduction (R) events, referenced to Fc+/0.

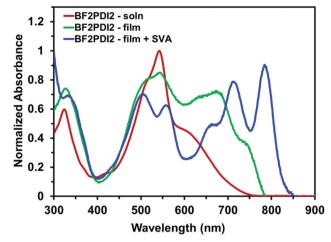


Fig. 3 UV-vis-nIR absorption profile of BF₂PDI₂ in CHCl₃ solution (red), thin-film spin-cast from o-dichlorobenzene (green), and the same thinfilm solvent vapor annealed from CHCl₃ (blue).

assigned. On the other hand, the first and fourth reductions $(E_{1/2} = -0.85 \text{ and } -1.96 \text{ V vs. Fc}^{+/0})$ may be attributed to **BF₂fz** core, while the second and third reduction events ($E_{1/2} = -1.32$ and $-1.60 \text{ V} \text{ vs. Fc}^{+/0}$) may be attributed to N-annulated PDI as the current passed during these redox processes was $2 \times$ larger. By DFT calculations, the first oxidation event appeared to be delocalized across the entire molecule, while the first reduction event was largely centered on the BF₂fz core (Fig. S15, ESI†). The predicted HOMO-LUMO energy levels correlate well with the observed CV data. Together, these data strongly suggest that the BF₂fz core has a higher electron affinity than the N-annulated PDI endcaps, which served to lower the LUMO energy level of BF₂PDI₂, when compared to related compounds with different cores.²¹

The optical properties of BF₂PDI₂ were measured by UV-visiblenearIR absorption spectroscopy (UV-vis-nIR). The UV-vis-nIR profile of BF₂PDI₂ in solution exhibited a broad absorbance between 450-750 nm, with an absorption maximum (λ_{max}) at 543 nm attributable to the PDI endcaps and a low energy shoulder just beyond 600 nm attributed to the BF2fz core (Fig. 3). By overlaying the optical spectra of BF₂PDI₂ with its constituent fragments (BF₂Ace₂ and PDIN-EH, Fig. S11, ESI†), it was revealed that the low energy shoulder attributed to BF2fz core was red-shifted in BF2PDI2, emphasizing the extension of π -conjugation induced by the PDI endcaps. When spin-cast into a thin-film, the optical profile of BF₂PDI₂ underwent several notable changes. Beyond the slightly red-shifted onset of absorption (λ_{onset}) , the broadening of λ_{max} was also accompanied by an appreciable increase in the intensity of the low-energy shoulder, consistent with enhanced electron delocalization along the π -conjugated backbone.²¹

In the past, we have shown that post-deposition solvent vapor annealing (SVA) can induce structural order within the solid-state morphology of similar PDI-core-PDI materials. 11,28-31 Exposure of the BF₂PDI₂ thin-film to CHCl₃ solvent vapors caused the once broad solid-state optical profile to resolve into two distinct absorption regions. The PDI-based λ_{max} region was split into two peaks (λ = 508 and 560 nm), while the low-energy shoulder

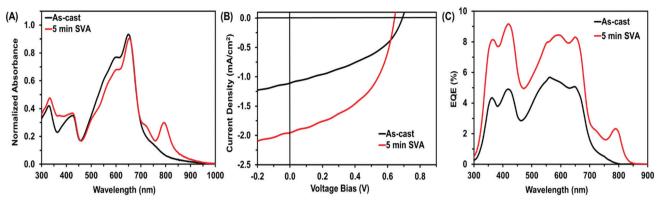


Fig. 4 Optical absorption profile (A), J-V curves (B), an external quantum efficiency plots (C) comparing the effects of solvent vapor annealing with CHCl₃ on ternary blend OPV devices with a FBT: BF₂PDI₂: PC₆₁BM (1:1:0.5) active layer at 10 mg mL⁻¹ total concentration

developed fine structure (λ_{max} = 790 nm) that extended well into the near-IR (λ_{onset} = 850 nm). This behaviour is strongly indicative of molecular reorganization and/or aggregation of BF₂PDI₂ into ordered domains with strong intermolecular electronic-coupling in the solid-state.32-34

OPV devices

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The NFA capabilities of BF2PDI2 were assessed by constructing some proof-of-concept OPV devices with the following inverted architecture: ITO/ZnO/ternary active layer/MoOx/Ag (full experimental details in ESI†). A ternary active layer comprised of electron-donor polymer PPDT2FBT (FBT), PC₆₁BM, and BF₂PDI₂ was employed for OPV devices. FBT was selected because of its complementary absorption with the solvent vapor annealed film of BF₂PDI₂ (Fig. S16, ESI†). PC₆₁BM was added to this ternary blend to assist with charge mobility. 35 Using a FBT: BF2PDI2: PC₆₁BM blend ratio of (1:1:0.5) at 10 mg mL⁻¹ total concentration, the best OPV devices achieved an open circuit voltage $(V_{\rm oc})$ of 0.69 V, a short circuit current $(J_{\rm sc})$ of 1.1 mA cm⁻², and a fill factor (FF) of 41%, leading to a power conversion efficiency (PCE) of 0.3% (Table S1, ESI†).

Post-deposition treatment of these films with CHCl₃ SVA resulted in the desired optical profile shift of BF₂PDI₂, giving the OPV devices a near-panchromatic absorbance (Fig. 4A). Device performance was maximized after 5 min of CHCl₃ SVA, where both the J_{sc} and FF increased to 1.9 mA cm⁻² and 49%, respectively, leading to a 2-fold increase in PCE to 0.6% (Fig. 4B). Longer SVA treatments led to OPV performances similar to ascast devices (Fig. S17 and Table S2, ESI†). This phenomena has previously been observed with our N-annulated PDI-core-PDI materials and was attributed to over-annealing of the active layer.11,28

To better understand the observed enhancement in OPV performance for FBT:BF₂PDI₂:PC₆₁BM (1:1:0.5) ternary blends that were SVA using CHCl₃ for 5 min, photoluminescence (PL) and external quantum efficiency (EQE) experiments were performed. PL measurements of the ternary blend showed efficient quenching of the FBT polymer before and after SVA treatment (Fig. S18, ESI†). Analysis of the EQE spectra, the

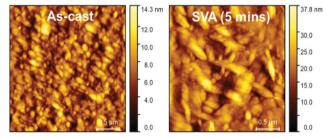


Fig. 5 Atomic force microscopy height images of ternary blend OPV devices with an FBT: BF₂PDI₂: PC₆₁BM (1:1:0.5) active layer at 10 mg mL⁻¹ total concentration

measured photocurrent clearly displays contributions from all components of the ternary blend (Fig. 4C). Following SVA treatment, photocurrent generation was extended well into the near-IR (λ_{max} = 790 nm), clearly emphasizing the increased contribution from the new NFA material, BF₂PDI₂.

Surface morphology differences between the as-cast and SVA treated devices were also analyzed by atomic force microscopy (AFM). AFM height images showed significant differences between the as-cast and SVA treated devices (Fig. 5), as the root-mean square surface roughness of the films increased from 1.9 nm to 4.8 nm. X-ray diffraction (XRD) measurements of ternary blend films before and after CHCl3 SVA failed to show any distinguishing features that could be attributed to BF₂PDI₂ (Fig. S19, ESI†).³⁶ Together, these data suggest that SVA enhanced OPV performance by inducing BF₂PDI₂ aggregation into phase separated domains, rather than crystallization.

Conclusions

In conclusion, we have synthesized and fully characterized a first-of-its-kind NFA containing a BF2fz core. The use of acetylene-linkers enabled through-conjugation between the BF₂fz core and the PDI endcaps, leading to an altered LUMO energy level that resulted in near-panchromatic absorption for BF₂PDI₂. The new compound is a rare example of an acceptoracceptor-acceptor type π -conjugated architecture. Treatment of solid-state BF2PDI2 with CHCl3 SVA resulted in a significant bathochromic shift of the optical spectra, leading to near-IR absorption. The overall performance in a series of proof-ofconcept OPV devices, where BF2PDI2 was used as an NFA in a ternary blend system, was improved 2-fold by SVA treatment of the active layer. The increase in PCE could be mainly attributed to favorable aggregation of BF₂PDI₂ in the active layer which increased the generated photocurrent. This notion was supported by EQE and AFM measurements. This work demonstrates the utility of the BF₂fz organic dye to construct narrow bandgap π -conjugated materials for use in organic electronics and provides the groundwork for further development.

Conflicts of interest

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There are no conflicts to declare.

Acknowledgements

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