



# Materials and Device Architectures for Organic Solar Cell Applications

## Cooperative Research and Development Final Report

**CRADA Number: CRD-09-355**

NREL Technical Contact: Andrew Ferguson

**NREL is a national laboratory of the U.S. Department of Energy  
Office of Energy Efficiency & Renewable Energy  
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Contract No. DE-AC36-08GO28308

**Technical Report**  
NREL/TP-5900-72590  
October 2018



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**Technical Report**  
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October 2018

National Renewable Energy Laboratory  
15013 Denver West Parkway  
Golden, CO 80401  
303-275-3000 • [www.nrel.gov](http://www.nrel.gov)

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## Cooperative Research and Development Final Report

In accordance with requirements set forth in the terms of the CRADA agreement, this document is the final CRADA report, including a list of subject inventions, to be forwarded to the DOE Office of Science and Technical Information as part of the commitment to the public to demonstrate results of federally funded research.

**Parties to the Agreement:** University of Queensland

**CRADA number:** CRD-09-355

**CRADA Title:** Materials and Device Architectures for Organic Solar Cell Applications

**Joint Work Statement Funding Table showing DOE commitment:**

Estimated Costs	NREL Shared Resources a/k/a Government In-Kind
Year 1	\$300,000.00
Modification #1 - Modification #6	\$300,000.00 \$450,000.00
TOTALS	\$1,050,000.00

### **Abstract of CRADA Work:**

The Centre for Organic Photonics & Electronics (COPE) at UQ has been successful in acquiring funding from the Australian Center for Advanced Photovoltaics (ACAP), which is the Australian partner of the Australia-US Institute for Advanced Photovoltaics (AUSIAPV).. NREL is a key partner in AUSIAPV (along with other US research institutions (ASU, Sandia NL, LBNL, Stanford, Georgia Tech and UCSB). NREL will collaborate with COPE to advance the science and technology of organic materials for solar cell applications. COPE has significant programs in the development and understanding of new materials for organic optoelectronic applications and, in particular, for thin film bulk heterojunction (BHJ) solar cells and Organic Photodiodes (OPDs). NREL has significant established research programs in all aspects of organic photovoltaic technology and has well-developed expertise in the area of materials characterization, photocarrier transport measurements as well as device fabrication, scale-up and stability. This collaboration will combine the expertise of the two institutions to work towards demonstrating the scalability of organic materials for BHJ and OPD applications by measuring the fundamental properties of the materials as a function of processing and evaluating their performance and stability in photovoltaic devices under simulated illumination conditions. Currently, both NREL and UQ have individual organic material development and processing efforts and have independently demonstrated several prototype materials and device architectures as promising candidates for organic solar cells and OPDs. This CRADA will align these efforts building upon strengths of the two institutions to further design and demonstrate materials and device architectures to advance organic BHJ solar cells and OPDs. It should also be clearly noted

that we foreshadow possible expansion of collaborative research activities between COPE and NREL, which currently lie outside the scope of the current statement of work, which will be amended as required by mutually executed written modifications to this CRADA.

### **Summary of Research Results:**

In its entirety (including amendments) the project included three Tasks/Themes: (1) Dendritic sensitizers for DSSCs; new conjugated dendrimers and small molecules for (2) bulk-heterojunction organic photovoltaics (OPV) and (3) Organic Photodetectors. The strong materials synthesis capabilities at UQ were combined with unique spectroscopic characterization and device expertise at NREL to create the synergy reflected in this CRADA. Work carried out on the individual tasks is outlined below.

### **Task Descriptions and Estimated Completion Dates**

This collaboration has a time period of three years (lifetime of OSCA) with the tasks for the first 18 months being described below. The tasks for the second 18 months will be determined at the first-year review by the OSCA management committee on which both NREL and COPE are represented. The costing for the first 18 months is shown in the funding table with a concomitant allocation of funding reserved for the second 18 months as per the NIRAP application.

### **Theme 1: Sensitizer dyes for DSSCs:**

#### **Original CRADA: Dendritic sensitizers for DSSCs.**

COPE has developed a range of ruthenium complex cored dendrimers as sensitizers for DSSCs. NREL has long-standing expertise in synthesis of high-quality TiO<sub>2</sub> particles and nanotubes, the deposition of TiO<sub>2</sub> nanostructured films with controlled properties, the fabrication of DSSCs and the characterization of the dynamics of photocarrier transport and recombination. The NREL group, however, has limited capability to develop new sensitizers. This task aims to combine the areas of expertise of the UQ and NREL groups in order to advance the science and technology of DSSCs. UQ will provide the developed sensitizers for NREL to undertake the following measurements:

- Effect of the dendritic dye structure on the injection and transport dynamics of photocarriers in nanostructured TiO<sub>2</sub> films
- Effect of the dendritic structure on the recombination kinetics / parasitic reactions of photocarriers in TiO<sub>2</sub> nanostructured films and potentially through electrolyte back reactions
- Effect of the dendritic structure on the efficiency of DSSCs. Testing and performance verification will be done on laboratory-scale devices (<1cm<sup>2</sup>)
- Effect of the dendritic structure on the stability and reliability of DSSCs
- Effect of the morphology of the TiO<sub>2</sub> film (nanotube vs nanoparticle) on the adsorption of the dendritic dyes.

### **CRADA Mod1: Type II sensitizers for DSSCs.**

COPE has developed a range of catechol dyes as sensitizers for DSSCs. This task aims to combine the areas of expertise of the UQ and NREL groups in order to advance the science and technology of DSSCs. UQ will provide the developed sensitizers for NREL to undertake the following measurements:

- Effect of the catechol dye structure on the injection and transport dynamics of photocarriers in nanostructured TiO<sub>2</sub> films
- Effect of the catechol structure on the recombination kinetics / parasitic reactions of photocarriers in TiO<sub>2</sub> nanostructured films and potentially through electrolyte back reactions
- Effect of the catechol structure on the efficiency of DSSCs. Testing and performance verification will be done on laboratory-scale devices (<1 cm<sup>2</sup>).

A number of novel dendritic and catechol-based sensitizers were tested with no remarkable outcomes. This part of the work was not pursued beyond the initial stages of the project and no joint publications resulted from it. The efforts of the CRADA moved to OPV, given its higher R&D impact (at the time).

### **Theme 2: Novel dendrimers and small molecules for bulk heterojunction organic photovoltaics**

#### **Original CRADA: Characterization of fundamental dendrimer properties for BHJ organic photovoltaic (OPV) applications.**

COPE and NREL have had independent programs on the synthesis and characterization of dendrimers for BHJ OPV applications. COPE has strong expertise in an array of synthetic routes for dendritic structures and is currently developing the capability to characterize these structures in terms of film morphology and electronic properties. NREL has expertise on the measurement of device-relevant properties such as the spectroscopic characterization of dendrimers in solution and in films as well as the transient photoconductivity of films and devices based on dendrimers. In addition, NREL is the premier laboratory for validation of photovoltaic device performance in the US and is one of the leading institutions in an international effort to standardize OPV efficiency and reliability measurements. The NREL team is currently expanding its synthetic capabilities to include more complex dendritic structures that are specifically designed for BHJ OPV applications. This task aims to combine the areas of expertise of the two institutions in order to a) assist NREL's synthetic efforts and b) combine different characterizations of morphology (COPE) and exciton and photocarrier dynamics (NREL) in order to advance the science and technology of BHJ OPV devices based on dendrimers.

#### **CRADA Mod1: Acceptors for BHJ organic photovoltaic (OPV) applications.**

This task aims to combine the areas of expertise of the two institutions. UQ is developing new electron acceptors for utilisation with donors from Phase I for BHJs. The program will combine

different characterizations of morphology (COPE) and exciton and photocarrier dynamics (NREL) in order to advance the science and technology of BHJ OPV devices.

### **CRADA Mod6: Materials and devices with BHJ solar cells.**

NREL will conduct photocarrier generation and decay analysis in new material systems for BHJ solar cells. NREL will also characterize the optical properties of the materials. NREL and COPE will work together to measure carrier mobilities. With the experimental information available, the scaling of these materials for device applications will be considered to select the most promising candidates. Device prototypes, including larger area ( $>1 \text{ cm}^2$ ) devices will be fabricated and characterized.

The research defined in the three joint work statements (Original, Mod1, and Mod6) was broadly categorized into the following experiments:

- NREL will perform Microwave conductivity measurements on new donor and acceptor materials in thin film BHJ solar cells produced at COPE – In particular, NREL will provide photoconductivity action spectra and carrier lifetime measurements to evaluate the quality of active layers that utilize the molecules. The measurements will be correlated with time-of-flight and/or field effect mobility measurements done collaboratively between NREL and COPE.
- COPE will perform morphology characterization on the BHJ films, utilizing its access to neutron scattering and synchrotron facilities subject to applications for facility/beam time being successful.
- NREL will perform Time-Resolved Photoluminescence and Photoluminescence Anisotropy spectroscopy in order to elucidate the exciton dynamics in the BHJ films.
- NREL will fabricate and validate the efficiency of prototype BHJ devices with new acceptor systems produced at COPE – device fabrication and testing to AM1.5G standards (efficiency and IPCE). Cross-correlation with the new OPV testing facility at COPE will continue validate that facility to accepted international standards.

Early work on dendritic donor materials indicated that the performance was significantly reduced compared to polymeric counterparts and, as such, these studies were not pursued beyond the initial stages of the project and no joint publications resulted from them. The efforts of the CRADA moved to investigating non-fullerene, small molecule-based electron acceptors for OPV, resulting in four joint publications between NREL and COPE (NREL Authors in **Bold**). These publications are identified and summarized below:

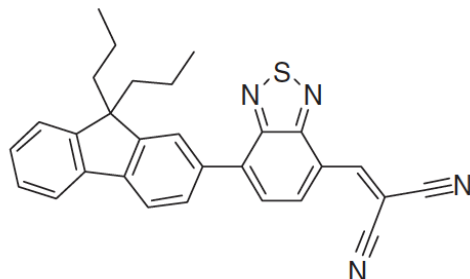
P. E. Schwenn, K. Gui, **A. M. Nardes**, K. B. Krueger, K. H. Lee, K. Mutkins, H. Rubinstein-Dunlop, P. E. Shaw, **N. Kopidakis**, P. L. Burn, P. Meredith,

“A Small Molecule Non-fullerene Electron Acceptor for Organic Solar Cells”

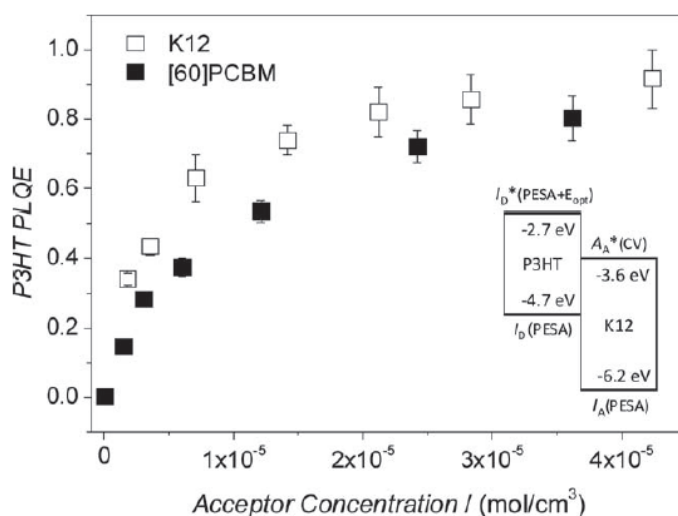
*Adv. Energy Mater.*, **1** (2011) 73-81. <https://dx.doi.org/10.1002/aenm.201000024>

Initial studies were performed on 2-[[7-(9,9-di-*n*-propyl-9*H*-fluoren-2-yl)benzo[*c*][1,2,5]thiadiazol-4-yl]methylene]malononitrile (K12), Fig. 1, which was synthesized

at COPE. Photoluminescence quenching experiments suggested that the K12 electron acceptor effectively quenched excitons generated in the prototypical conjugated polymer donor, poly(3-*n*-hexylthiophene-2,5-diyl) (P3HT), Fig. 2. These data indicated that K12 is even more effective than the prototypical fullerene electron acceptor, [C60]PCBM, at least at low acceptor concentrations.



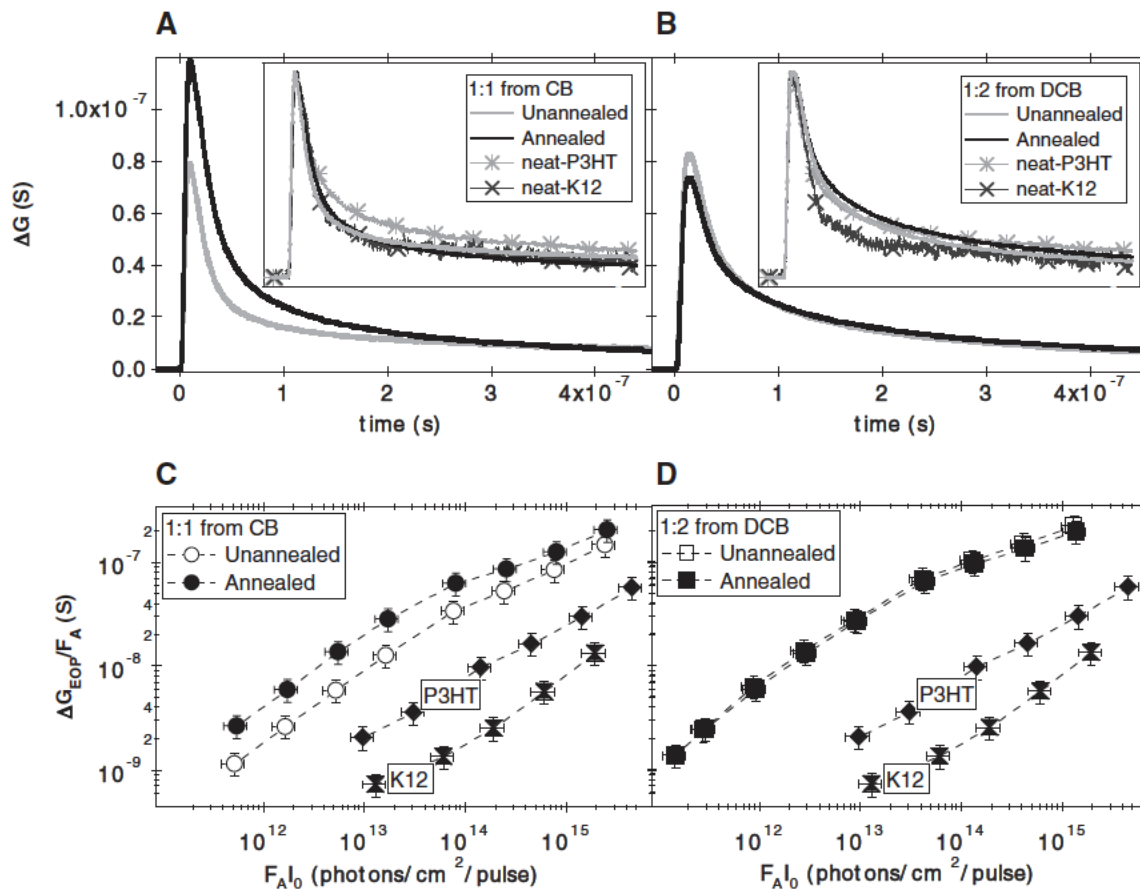
**Figure 1.** Chemical structure of K12.



**Figure 2.** Photoluminescence quenching efficiency of P3HT with various weight ratios of K12, compared to the prototypical fullerene electron acceptor, [C60]PCBM.

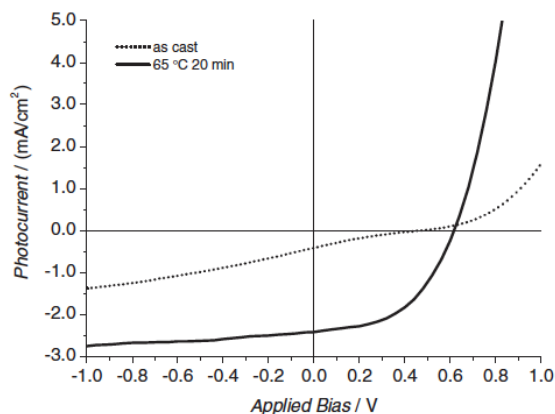
Flash-photolysis time-resolved microwave conductivity (TRMC) measurements of P3HT:K12 bulk heterojunctions (BHJs) indicated that the exciton quenching process resulted in the formation of mobile charge carriers, Fig. 3. The data in Fig. 3A and Fig. 3C suggest that post-deposition thermal annealing, at 65 °C for 20 minutes, improves the photoconductance of the BHJ deposited from chlorobenzene (CB). In contrast, the photoconductance of the BHJ deposited from the higher boiling point solvent ortho-dichlorobenzene (DCB) is unaffected by thermal annealing, Fig. 3B and Fig. 3D. We attributed this observation to differences in the BHJ morphology due to the different processing conditions: the reduced rate of DCB evaporation appears to allow for a more favourable morphology for efficient mobile charge carrier generation than for CB, although this difference can be overcome by a post-deposition thermal annealing process. However, the peak photoconductances measured at low incident photon flux are similar for the annealed BHJ deposited from CB (Fig. 3C) and the BHJs from DCB (Fig. 3D), suggesting that the short-range nanomorphology probed by the TRMC technique is similar for the two samples.





**Figure 3.** Photoconductance of P3HT:K12 blends with a weight ratio of 1:1 in CB (A & C) and 1:2 in DCB (B & D), as measured by time-resolved microwave conductivity. (A & B) Photoconductance transients measured at an incident photon flux of ca.  $2 \times 10^{15} \text{ cm}^{-2}$ . The insets show the normalized photoconductance transients and a comparison with neat films of P3HT and K12. (C & D) Peak photoconductance, normalized by the fraction of absorbed light ( $F_A$ ), as a function of photon flux for the same samples.

In contrast to the observations from TRMC measurements, photovoltaic devices prepared from DCB perform better than those prepared from CB, resulting in an optimized power conversion efficiency (PCE) of 0.73% for the device spun cast from DCB and annealed at 65 °C for 20 minutes (Fig. 4). The observation of optimum performance after annealing at 65 °C was attributed to the increased molecular order in the K12 phase, due to annealing above the K12 glass transition temperature.



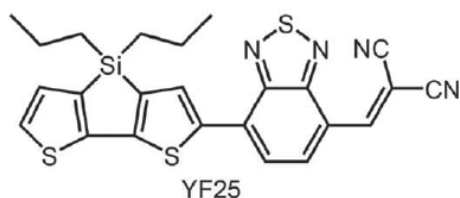
**Figure 4.** Photovoltaic device performance of a P3HT:K12 bulk heterojunction solar cell with a weight ratio of 1:2 in DCB.

Y. Fang, A. K. Pandey, **A. M. Nardes**, **N. Kopidakis**, P. L. Burn, P. Meredith,

“A Narrow Optical Gap Small Molecule Acceptor for Organic Solar Cells”

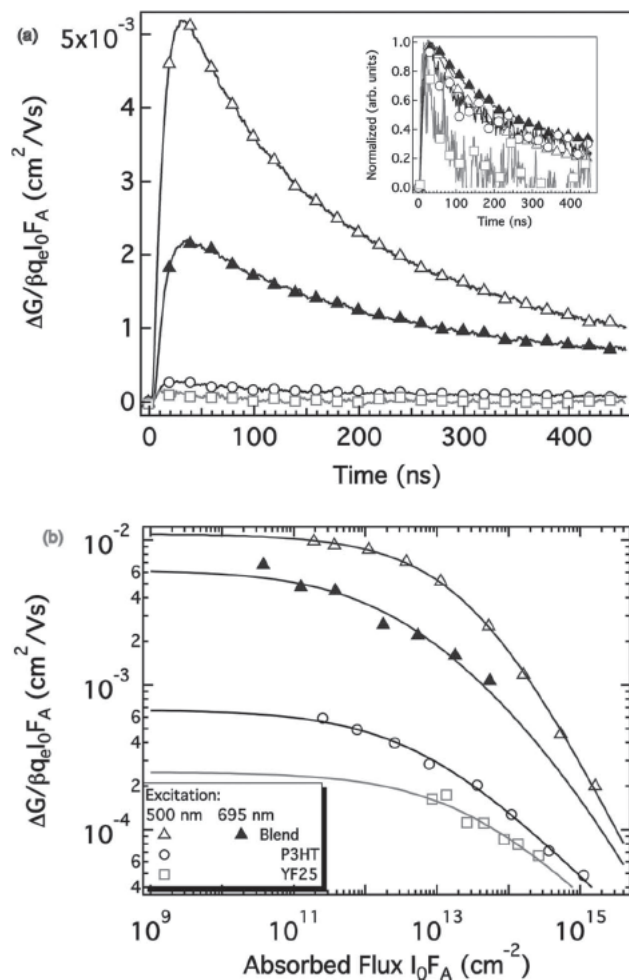
*Adv. Energy Mater.*, **3** (2013) 54-59. <https://dx.doi.org/10.1002/aenm.201200372>

Motivated by the promise shown by K12, COPE developed a similar non-fullerene, small molecule electron acceptor, 2-[[7-(4,4-di-*n*-propyl-4*H*-silolo[3,2-*b*:4,5-*b'*]dithien-2-yl)benzo[*c*][1,2,5]thiadiazol-4-yl]methylene]malononitrile (YF25), Fig. 5, with a red-shifted absorption spectrum. The change in from the (9,9-di-*n*-propyl-9*H*-fluoren-2-yl) moiety to (4,4-di-*n*-propyl-4*H*-silolo[3,2-*b*:4,5-*b'*]dithien-2-yl) was made in an effort to broaden the spectral response of BHJ solar cells prepared with poly(3-*n*-hexylthiophene-2,5-diyl) (P3HT).



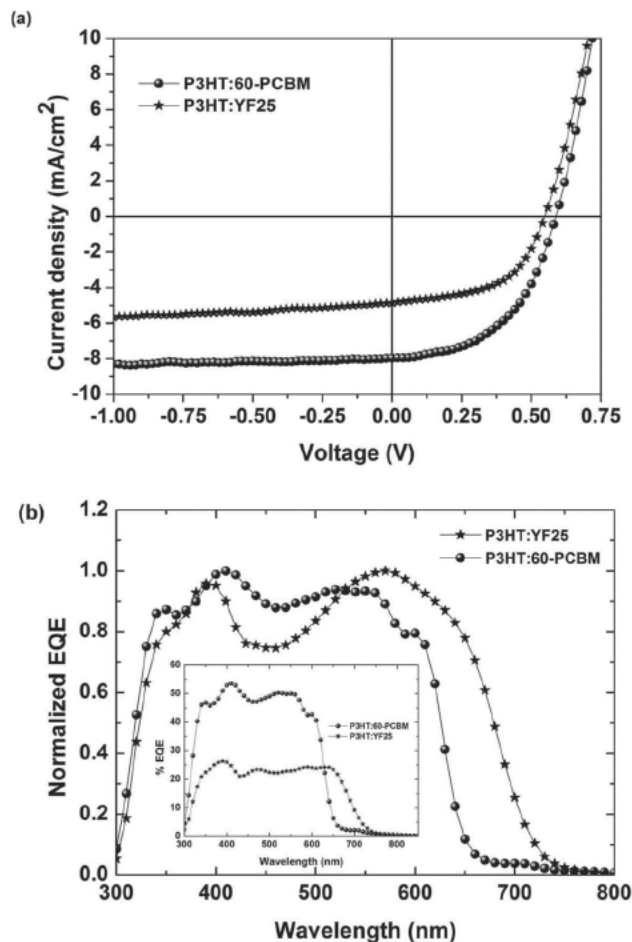
**Figure 5.** Chemical structure of YF25.

As for the P3HT:K12 BHJ, flash-photolysis TRMC measurements of P3HT:YF25 BHJs indicated that the exciton quenching process resulted in the formation of mobile charge carriers, Fig. 6. However, a number of differences are observed for the P3HT:YF25 BHJ: (1) the carrier lifetime is significantly improved (and longer than measured for the neat P3HT polymer) for YF25 than K12, (2) YF25 enables a larger enhancement (ca. 30X) of the peak photoconductance than K12, relative to the neat P3HT polymer, (3) photoexcitation of the YF25 results in mobile carrier generation, due to photoinduced hole transfer (PHT), and (4) the yield of mobile carriers is reduced for photoexcitation of YF25 compared to photoexcitation of P3HT.



**Figure 6.** (a) Photoconductance decay transients measured by TRMC for a P3HT:YF25 (1:1.5, by wt) BHJ, neat P3HT, and neat YF25 excited at 500 and 695 nm at a fixed photon flux of  $\sim 1 \times 10^{13} \text{ cm}^{-2}$ . The inset shows the intensity-normalized photoconductance decays for the same data. (b) The photoconductance values as a function of photon flux. The photoconductance values are normalized by the absorbed photon flux ( $I_0 F_A$ ), the microwave cavity geometry factor ( $\beta$ ), and the elementary charge ( $q_e$ ).

The improved photoconductance properties observed for the P3HT:YF25 BHJ compared to P3HT:K12 is manifest in an improved photovoltaic device performance, resulting in an optimized PCE of 1.43%. Figure 7b confirms that the spectral response of the photovoltaic device is extended by blending P3HT with YF25. However, BHJs prepared with YF25 still underperform those prepared with the prototypical fullerene electron acceptor, [C60]PCBM. This observation is attributed to formation of extended YF25 domains, thereby limiting effective carrier extraction from devices, as compared to [C60]PCBM.



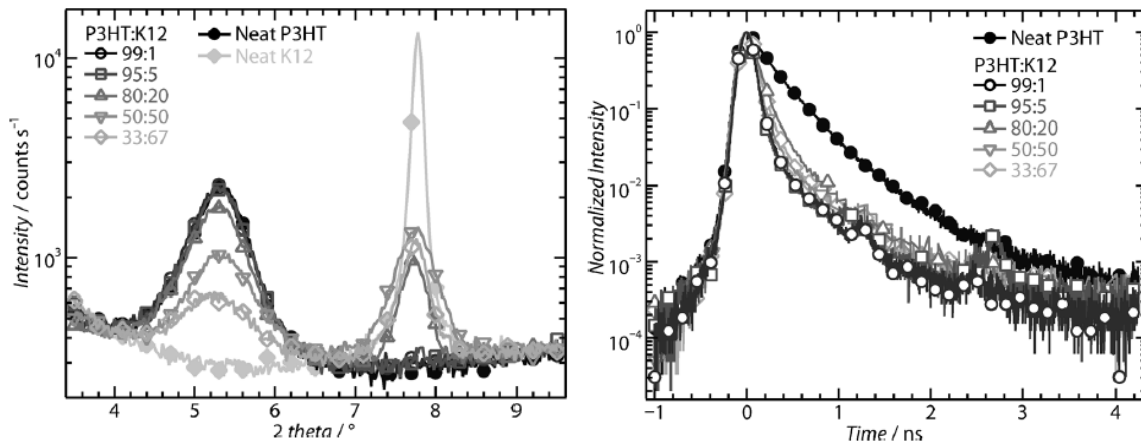
**Figure 7.** Comparison of photovoltaic device performance of P3HT:[C60]PCBM and P3HT:YF25 solar cells (representative of multiple devices). (a) Current density-voltage characteristics and (b) External quantum efficiency (EQE) measurements (main panel: normalised; inset: non-normalised).

A. M. Nardes, A. J. Ferguson, P. Wolfer, K. Gui, P. L. Burn, P. Meredith, N. Kopidakis,

“Free Carrier Generation in Organic Photovoltaic Bulk Heterojunctions of Conjugated Polymers with Molecular Acceptors: Planar versus Spherical Acceptors”

*ChemPhysChem*, **15** (2014) 1539-1549. <https://dx.doi.org/10.1002/cphc.201301022>

The comparison of P3HT:K12 and P3HT:[C60]PCBM BHJs from the first joint paper were investigated in more detail. Structural measurements, performed using X-ray diffraction, showed no evidence for pure K12 domain formation in the P3HT:K12 BHJs at K12 loadings lower than 20% by weight, whereas ordered K12 domains are formed at loadings greater than 20% (Fig. 8; left). However, photoluminescence decays, measured using time-correlated single-photon counting (TCSPC), suggest that quenching of P3HT excitons is efficient even at low K12 loadings (Fig. 8; right).



**Figure 8.** (left) Powder X-ray diffraction patterns and (right) Photoluminescence decays, normalized to the peak intensity, as a function of K12 loading, of P3HT:K12 bulk heterojunction films deposited onto quartz substrates.

Comparison of the data extracted using TCSPC and TRMC suggests that the P3HT-K12 interface is an extremely efficient exciton quenching interface, but that the yield of long-lived mobile carriers is rather low (Table 1). These combined observations are attributed to the formation of an intimately mixed, glassy (amorphous) P3HT-K12 phase at low loadings, where effective carrier percolation to the pure P3HT and K12 phases is impeded by poor electron transfer between neighboring K12 molecules in the mixed P3HT-K12 phase.

Table 1. Photoluminescence lifetimes, exciton quenching efficiency, and free carrier generation yields.			
Sample <sup>[a]</sup>	$\tau$ [ps]	$Q_E^{[b]}$	$\varphi^{[c]}$
P3HT (100:0)	285	–	0.03
P3HT:K12 (99:1)	35	0.88	0.05
P3HT:K12 (95:5)	33	0.88	0.09
P3HT:K12 (80:20)	88	0.70	0.11 <sup>[d]</sup>
P3HT:K12 (50:50)	77	0.73	0.26 <sup>[d]</sup>
P3HT:K12 (33:67)	65	0.77	0.19 <sup>[d]</sup>

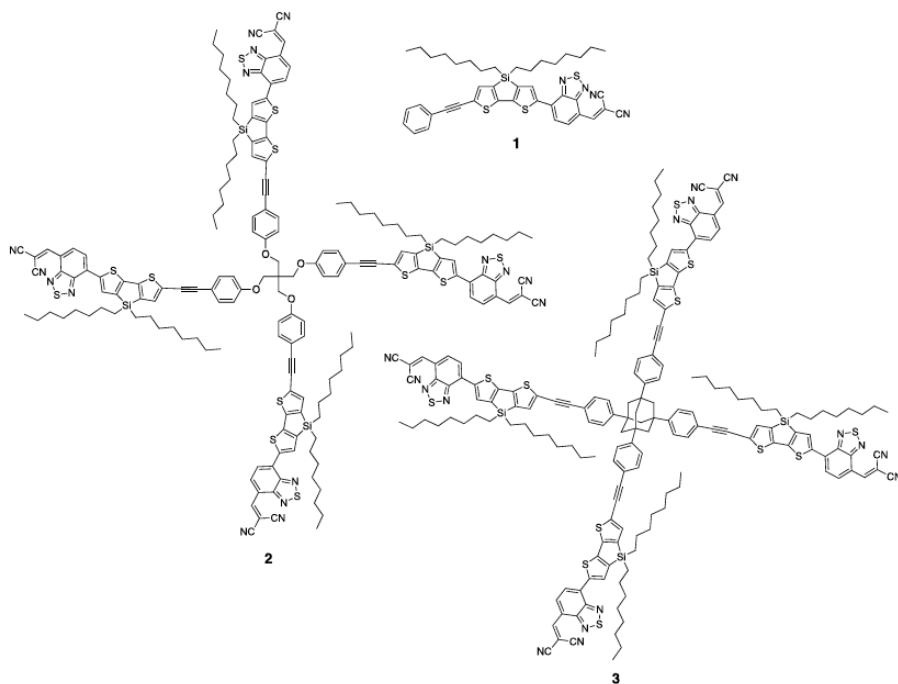
[a] Composition given in parentheses is the mass ratio. [b] Calculated from the photoluminescence lifetimes relative to the neat polymer, using Equation (1). [c] Calculated from the low-intensity (linear) yield-mobility product determined using TRMC, assuming only a contribution to the sum of mobilities from mobile holes in P3HT. [d] Upper limit of the free carrier generation yield per absorbed photon (see text).

Dani M. Stoltzfus, **Bryon W. Larson**, Nasim Zarrabi, Paul E. Shaw, Andrew J. Clulow, Hui Jin, Paul L. Burn, Ian R. Gentle, **Nikos Kopidakis**,

“Investigating charge generation in polymer:non-fullerene acceptor bulk heterojunction films”

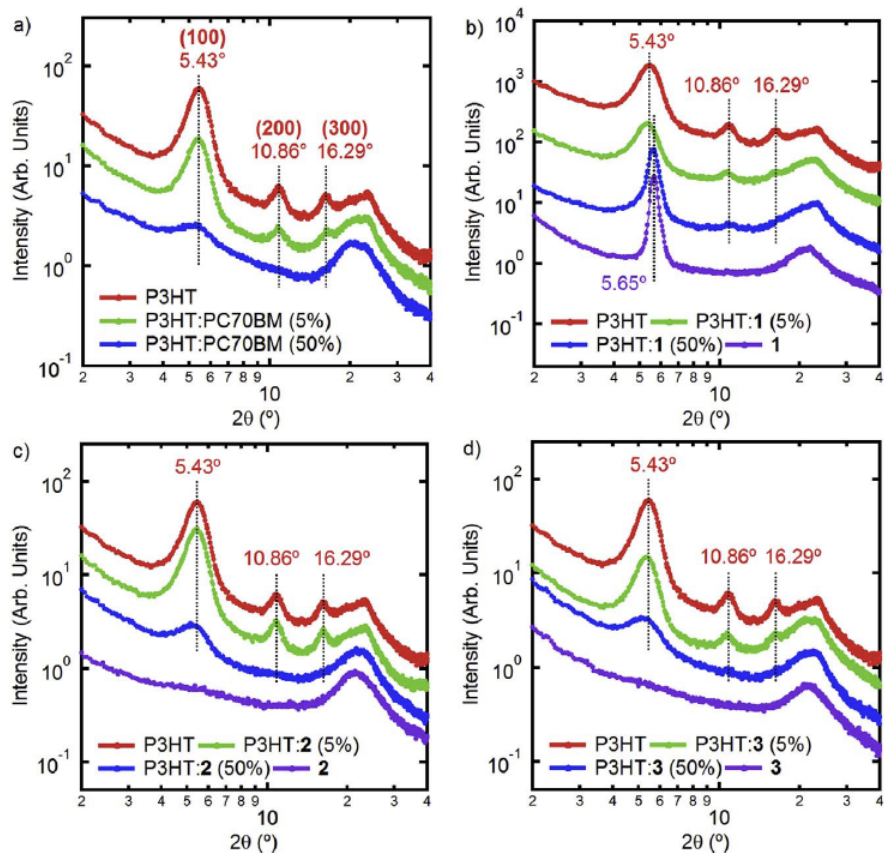
*Organic Electronics*, **55** (2018) 177-186. <https://dx.doi.org/10.1016/j.orgel.2018.01.017>.

The observations of (A) reasonable performance in BHJs formed from P3HT and YF25 and (B) improved long-lived mobile carrier generation in BHJs with non-planar electron acceptors, prompted a study of macromolecular (quasi-dendritic) electron acceptors formed from the active electronic YF25 chromophore (**1**) attached to inert cores based on a pentaerythritol (**2**) or an adamantyl (**3**) core moiety, Fig. 9.



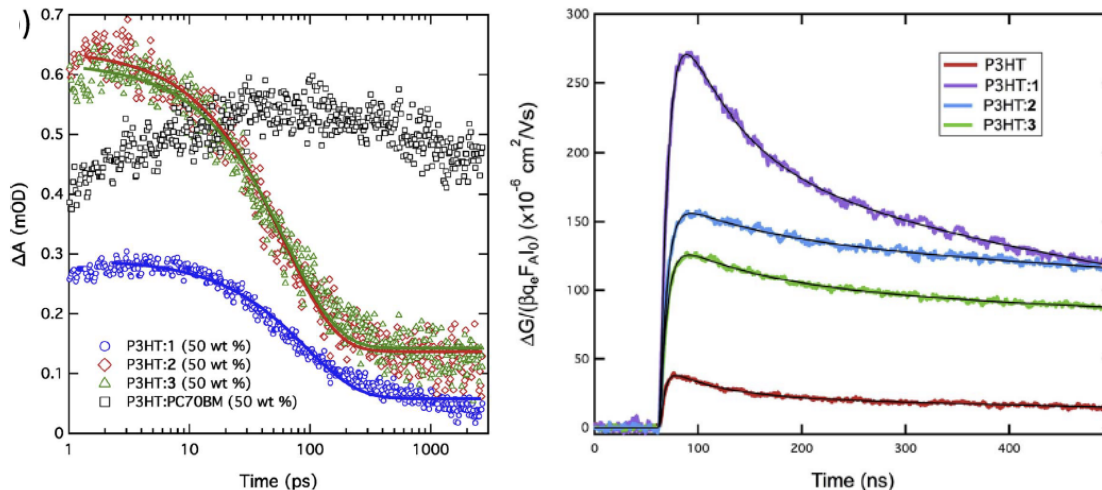
**Figure 9.** Chemical structures of the YF25-based electron acceptors.

Structural measurements of blends of P3HT with the three YF25-based electron acceptors, Fig. 10, indicate that the non-planar geometries afforded by attached the YF25 chromophore to the pentaerythritol (**2**) or an adamantyl (**3**) core moieties disrupts the formation of ordered electron acceptor domains observed in P3HT:1 blends.



**Figure 10.** Comparison of X-ray diffraction patterns of the neat materials and the P3HT:acceptor blends with a) PC70BM, b) **1**, c) **2**, d) **3**. Traces are offset vertically for clarity in order of increasing acceptor content from top to bottom.

To probe the influence of BHJ morphology/nanostructure we employed two time-resolved spectroscopic techniques: transient absorption spectroscopy (TAS) and time-resolved microwave conductivity (TRMC), Fig. 11. TAS measurements indicate prompt (sub-picosecond) formation of polarons in blends of P3HT and the YF25-based electron acceptors (Fig. 11; left), suggesting that carrier formation is non limited by diffusion, in contrast to the observation of polaron formation in blends of P3HT with the prototypical fullerene electron acceptor, [C70]PCBM. However, the rapid and efficient polaron formation is accompanied by a rapid polaron decay, with a large fraction of polarons disappearing within 1 ns, and kinetic analysis of the transients indicates a yield of long-lived polarons of ca. 20% for blends of P3HT with all three YF25-based electron acceptors. Assuming that the similar yields determined from TAS represent the initial mobile charge carrier yield detected by TRMC, the larger photoconductance observed for the P3HT:**1** BHJ suggests that the formation of extended (ordered) domains of **1** results in a detectable electron mobility contribution to the photoconductance. In contrast, the disorder introduced by attaching the YF25 chromophore to the pentaerythritol (**2**) or adamantyl (**3**) core moieties reduces this electron contribution. However, the photoconductance lifetimes are increased in the P3HT:**2** and P3HT:**3** blends, suggesting that the electrons are spatially separated from the holes in the P3HT.



**Figure 11.** (left) Polaron kinetics, measured using TAS, in P3HT blends with the electron acceptors [C70]PCBM, **1**, **2**, and **3**. (right) Photoconductance transients, measured using TRMC, of neat P3HT and P3HT blends with **1**, **2**, and **3**.

### **Theme 2 Summary:**

Overall, the collaboration between COPE and NREL has identified a number of important concepts that have had an impact on the field of bulk heterojunction organic photovoltaics (OPVs). These include:

- (1) Photoinduced hole transfer (PHT), following photoexcitation of strongly-absorbing electron-accepting chromophores, is a viable mechanism for photocurrent generation. By tailoring the electronic properties of these chromophores, the power conversion efficiency of OPV devices can be enhanced by improved spectral response.
- (2) Long-lived photocarrier generation is dependent on the morphology of the bulk heterojunction, particularly in the donor-acceptor mixed phase. As people have transitioned from fullerene-based electron acceptors to planar small molecules, the interplay between effective electronic coupling between acceptor chromophores and deleterious formation of extended electron acceptor domains needs to be achieved to maximize the yield of long-lived mobile charge carriers that can be extracted from the photovoltaic active layer.



### **Theme 3: Organic Photodiodes:**

This part of the project involved modifying an OPV absorber layer to create the best reported narrowband near-IR, visible-blind, organic photodetector (OPD) that operates without the use of optical filtering. COPE created and tested the photodetector device and NREL contributed to interpretation of device results and to the creation of the operational model of the device. Given the significance of the work (it was published in *Nature Communications*—see publication information below—and it became one of NREL's highlight publications at the time), an amendment to the CRADA was added to include photodetector work.

Ardalan Armin, Ross D. Jansen-van Vuuren, **Nikos Kopidakis**, Paul L. Burn, Paul Meredith, “Narrowband light detection via internal quantum efficiency manipulation of organic photodiodes”

*Nature Communications*, **6** (2015) 6343. <http://dx.doi.org/10.1038/ncomms7343>

### **CRADA Mod6: Organic Photodiodes**

NREL has a large database of organic structures that we will use to select materials for various OPD architectures. Candidate materials will be chosen based on optical gap and HOMO/LUMO energies. Materials will be synthesized at COPE and applied to OPDs with the goal of further developing narrowband and broadband OPD concepts.

This was the last stage of the project, and it did not progress beyond initial material selection prior to NREL's CRADA PI (Nikos Kopidakis) leaving his appointment at NREL.

### **Subject Inventions Listing:**

None

### **Report Date:**

12 August 2018

### **Responsible Technical Contact at Alliance/NREL:**

Andrew Ferguson

### **Name and Email Address of POC at Company:**

FAO Professor Paul Burn, [p.burn2@uq.edu.au](mailto:p.burn2@uq.edu.au)

### **DOE Program Office:**

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