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Thermally Activated n-Doping of Organic Semiconductors Achieved by N-heterocyclic Carbene Based Dopant

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Abstract: Molecular doping plays an important role in the modification of carrier density of organic semiconductors thus enhancing their optoelectronic performance. However, efficient n-doping remains challenging, especially due to the lack of strongly reducing and airstable n-dopants. Herein, an N-heterocyclic carbene (NHC) precursor, DMImC, is developed as a thermally activated n-dopant with the excellent stability in air. Its thermolysis in situ regenerates free NHC and subsequently dopes typical organic semiconductors. In sequentially doped FBDPPV films, DMImC doesn't disturb the π - π packing of the polymer and achieves good miscibility with the polymer. As a result, a high electrical conductivity of up to 8.4 S cm⁻¹ is obtained. Additionally, the thermally activated doping and the excellent air stability permit DMImC to be noninteractively co-processed with polymers in air. Our results reveal that DMImC can be served as an efficient n-dopant suitable for various organic semiconductors.

Molecular doping has been a powerful tool to improve the performance of organic semiconductor devices, in particular to modulate the electrical conductivity.^[1] The intrinsic organic conjugated materials are typically the insulators or semiconductors with low carrier density, molecular doping is usually employed to enhance their carrier density and conductivity.^[1a] P-doped conjugated polymers, such as poly[2,5bis(3-alkylthiophen2-yl)thieno(3,2-b)thiophene] (PBTTT) and poly(3,4-ethylenedioxythiophene) (PEDOT), have exhibited impressive electrical conductivities up to 1000 S cm⁻¹.^[2] However, n-doped conjugated polymers still show far more inferior conductivities than their p-type counterparts. One of the major limitations is the scarcity of air-stable n-dopants with strong ndoping ability. To realize an efficient n-doping, the ionization energy (IE) of dopants should be close to or lower than the electron affinity (EA) of organic semiconductors.^[3] However, the shallow IE of n-dopants results in their high sensitivity to air, which represents a major challenge to develop air-stable n-dopants for organic semiconductors.[3a, 4]

Recently, organic precursors have been proposed to circumvent the stability limitation of n-dopants.^[5] These precursors that liberate strong reducing species in situ upon external stimulus avoid using low IE n-dopants directly, increasing their air stability. Organic salts (including o-MeO-DMBI-I and PyB) exhibited good n-doping ability for organic semiconductors but were processed via vacuum deposition in most cases.^[6] Besides, a series of organic radical dimers and organic hydrides showed the excellent capability to n-dope a variety of organic

semiconductors.^[7] However, some of them are gradually oxidized or decomposed in the solution under ambient condition.^[8] Hence, more efforts are needed to develop air-stable yet strong reducing n-dopants for the development of n-doped organic semiconductor devices.

As strong σ donors, N-heterocyclic carbenes (NHCs) have become essential ligands in organic chemistry, with numerous applications in coordination chemistry and organocatalysis.^[9] Würthner and co-workers demonstrated free NHC reacted with chlorinated pervlene bisimide to yield delocalized zwitterion radical.^[10] This implies like hydride dopants that offer nucleophilic and electron-donating hydride,^[11] NHCs with strong nucleophilic and electron-donating ability can act as reactive species to dope organic semiconductors. Herein, we develop an air-stable NHC precursor, namely 1,3-dimethylimidazolium-2-carboxylate (DMImC) (Scheme 1a), as a thermally activated n-dopant. DMImC, an adduct of carbon dioxide and NHC, regenerates the corresponding NHC (1,3-dimethylimidazolylidene) via thermal activation, which subsequently in situ dopes organic semiconductors. DMImC exhibits excellent stability and good solubility in common polar solvents, facilitating its practical solution-processability. The small molecular volume of DMImC is beneficial for less perturbation of the polymer morphology and efficient incorporation into the underlying polymer layers in sequential casting.^[12] Further investigations demonstrate that NHC released from DMImC after activation can effectively n-dope representative n-type organic semiconductors (Figure S1) to obtain high electrical conductivity.

The reaction between 1-methylimidazole and dimethyl carbonate yielded the desired product, DMImC (Scheme 1b). Thermal gravity analysis (TGA) of DMImC afforded a moderate decarboxylation temperature of 159 °C (Figure S2). DMImC showed good air stability and solution stability at room temperature (Figure S3). Upon heating, DMImC reacted with N,N-dibutyl-1,6,7,12-tetrachloroperylene-3,4:9,10-

tetracarboxylic acid bisimide (PBI-Cl₄) to yield the PBI-centered radical anion (Scheme 1c), which was the same with the reported reaction of free NHC with PBI-Cl₄.^[10] The absence of hyperfine splitting in the electron paramagnetic resonance (EPR) spectrum and calculated spin density plot suggested that the unpaired electron was delocalized over the whole PBI skeleton (Figure S4). Above all, DMImC can be used as a promising thermally activated n-dopant to in situ generate NHC for n-doping.

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Scheme 1. a) Decarboxylation of DMImC to NHC and b) the synthetic approach to DMImC. c) The reaction between PBI-Cl₄ and DMImC under heating. A delocalized PBI-centered radical anion was yielded, in which the cationic imidazolium was introduced in the bay position of PBI-Cl₄. d) Chemical structure of polymer FBDPPV.

Due to the poor solubility of DMImC in the commonly used halogenated solvent, sequential casting was applied to prepare the DMImC-doped systems, in which DMImC was deposited from acetonitrile (an orthogonal solvent for organic semiconductors) onto the underlying polymer layer. FBDPPV was chosen as the host polymer for the subsequent studies (Scheme 1d), which was renowned for its high n-type field-effect mobility.^[13] The activation temperature of 160 °C was used as an external stimulus to trigger the decarboxylation of DMImC, followed by the n-doping process (Figure S5).

The doping process of FBDPPV with DMImC was firstly characterized by the UV-*vis*-NIR absorption spectra (Figure 1a). The intrinsic FBDPPV film displayed two absorption bands (band I: 600-900 nm; band II: 400-600 nm) and no absorption band around 1100 nm, which was attributed to the (bi)polaron absorption, was observed, accompanied by the bleaching of the neutral absorption. Polarons carrying unpaired electrons can be directly observed by the EPR spectra.^[1a] Doped FBDPPV film yielded an obvious paramagnetic signal with a characteristic electron g-factor of about 2.0039 (Figure 1b), indicating the generation of polarons. All these results suggest that NHC released in situ from DMImC can efficiently dope FBDPPV.

N(1s) X-ray photoemission spectroscopy (XPS) showed that after the thermal activation of DMImC, a quadrivalent nitrogen signal from the dopant cation arose at 402.7 eV (Figure S6), which was distinct from the original lactam nitrogen signal of FBDPPV (400.9 eV). The peak area ratio between these two peaks was used to monitor the DMImC incorporation upon sequential casting. DMImC with concentrations of 0.4, 0.6 and 1.0 mg mL⁻¹ in casting solution resulted in 0.36, 0.40 and 0.52 dopants per repeat unit (Figure 1c), respectively. In a word, the higher DMImC concentration permitted more DMImC to infiltrate into FBDPPV films and offered an improved doping level. This conclusion was also supported by the increased carrier density and Fermi level shift in doped FBDPPV films. The AC Hall effect measurements

showed that after doping, FBDPPV presented electron density of 2.33×10^{20} cm⁻³ at DMImC concentration of 0.4 mg mL⁻¹ (Figure 1c). Further increasing DMImC concentration to 1.0 mg mL⁻¹ led to an increased carrier density of 7.26×10^{20} cm⁻³. Based on ultraviolet photoelectron spectroscopy (UPS) measurement, the Fermi level of doped FBDPPV films shifted toward the lowest unoccupied molecular orbital (LUMO) (Figure 1d), confirming the effective occurrence of n-doping.^[11] When the concentration of DMImC reached 0.6 mg mL⁻¹, the Fermi level shift seemingly saturated, which perhaps arose from the pinning effect.^[14] Hence, we prove that the doping level of FBDPPV film can be controlled by varying the DMImC concentration in the casting solution.



Figure 1. a) UV-*vis*-NIR absorption spectra and b) EPR spectra of the intrinsic and doped FBDPPV films after thermal activation of DMImC. c) Molar fractions of dopant relative to FBDPPV repeat unit (calculated from the N(1s) XPS results) and the carrier density measured by the AC Hall effect as a function of DMImC concentration in the casting solution; d) UPS spectra around the secondary electron cutoff (SECO) regions of FBDPPV films.

To demonstrate the practical application of DMImC in organic electronics, electrical conductivity (σ), Seebeck coefficient (S) and power factor ($PF = S^2 \sigma$) of doped FBDPPV films were measured (Figure 2a and 2b). Upon doping, the electrical conductivity of FBDPPV dramatically increased with a maximal film conductivity of 8.4 \pm 0.6 S cm⁻¹ at the DMImC concentration of 0.5 mg mL⁻¹. Additionally, a high conductivity was also obtained in FBDPPV films doped by another NHC based n-dopant (DPImC) with Nisopropyl substituent (Figure S9), indicating that the strategy of thermally activated NHC based n-dopant was universally applicable. At DMImC concentration of 0.4 mg mL⁻¹, doped FBDPPV exhibited a negative Seebeck coefficient of -182 µV K⁻¹ and the coefficient further decreased with higher DMImC concentrations. Due to the high electrical conductivity and moderate Seebeck coefficient, an optimized power factor as high as 16 $\mu W~m^{\text{-1}}~K^{\text{-2}}$ at room temperature was achieved. The conductivities of another two famous n-type semiconductors N2200 and PCBM with higher LUMO levels than FBDPPV were

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also investigated to demonstrate the doping capability of DMImC (Figure 2c). After doping, N2200 and PCBM presented the maximum conductivities of $(5.4 \pm 0.6) \times 10^{-3}$ S cm⁻¹ and 0.10 \pm 0.02 S cm⁻¹, respectively. The conductivities of DMImC-doped systems were among the highest reported values doped by the best n-dopants nowadays (Figure S1 and S12), assuring that DMImC was a high-performance n-dopant for commonly used n-type organic semiconductors.

In most cases, the n-doped organic semiconductor devices were prepared in an inert atmosphere, increasing the complexity of device fabrications. The n-doping by thermally activated dopants involves a two-step process, deposition and postdeposition annealing.^[15] Given the excellent air stability and noninteraction with semiconductors before heating, DMImC can be noninteractively sequentially deposited onto FBDPPV films in air. The air stability of the n-doped films, which is seriously affected by H₂O or O₂ in air, is more dependent on the properties of n-type semiconductors, especially their electron affinity and crystallinity of polymer films.^[16] Since the n-doped FBDPPV thin films were sensitive to air (Figure S13),[7h] the subsequent annealing of doped FBDPPV thin films was performed in an inert atmosphere. FBDPPV films sequentially deposited with DMImC in air showed a maximal conductivity of 7.0 ± 0.7 S cm⁻¹ (Figure 2d), which was comparable to those prepared under inert gas. The combination of DMImC and post-deposition annealing provide the feasibility of deposition and handling the n-doped films in air, e.g., lithography and spin coating.



Figure 2. Thermoelectric performance characterization. a) Electrical conductivity, Seebeck coefficient and b) power factor of doped FBDPPV with various DMImC concentrations. c) Conductivities of doped N2200 and PCBM with various DMImC concentrations. d) Conductivities of doped FBDPPV films sequentially deposited with DMImC in air.

Grazing incidence wide-angle X-ray scattering (GIWAXS) was performed to analyze the solid-state microstructures of doped FBDPPV films. When the concentration (1.0 mg mL⁻¹) of DMImC far exceeded the optimum concentration for obtaining the maximal conductivity, the signs of π - π packing (010) and multiple

scattering of lamellar packing were still clearly observed (Figure 3b). Along with the increased DMImC concentrations, the lamellar packing distance of FBDPPV gradually increased from 36.5 Å to 38.3 Å, but the π - π packing distance stayed at 3.44 Å (Figure S14). The unchanged π - π packing distance which played a vital role in interchain carrier transport prevented the Hall mobility of doped films from a dramatic decrease as the concentration of DMImC increased (Figure S15b). The surface morphology of doped FBDPPV films was investigated by atomic force microscopy (AFM). The doped FBDPPV films displayed uniform morphology and fiber-like networks, which were the same as those undoped films. Moreover, no aggregated particles were observed on the surface of annealed films even at the high DMImC concentration of 1.0 mg mL⁻¹. The homogeneous morphology of doped FBDPPV films indicated the good miscibility between dopant and polymer, which was beneficial to high doping efficiency.^[17] Since the electrical conductivity depends on both mobility and carrier density, the undisrupted π - π packing and good miscibility contribute to the high electrical conductivity of doped FBDPPV.



Figure 3. Microstructure and morphology study of the intrinsic and doped FBDPPV films. a,b) GIWAXS images and c,d) surface topography mappings by AFM.

In summary, we have developed an air-stable thermally activated NHC based n-dopant, DMImC, for solution-processed n-doping. This thermally activated n-dopant provides several advantages: (1) simultaneously satisfying the requirement of excellent air stability and high n-doping efficacy; (2) easily triggered n-doping process by thermal activation; and (3) avoiding the formation of polymer/dopant ions when mixed directly and allowing to be noninteractively deposited with polymers even in air. Contribution from the good miscibility with polymer and unchanged π - π stacking distance in sequentially doped FBDPPV films benefit to high doping efficiency and efficient interchain carrier transport, which results in a high film conductivity. Additionally, FBDPPV films sequentially deposited with DMImC in

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air showed a maximal conductivity compared with those prepared under inert gas. From these results, we conclude that DMImC could be a high-performance n-dopant for various organic semiconductors applied in organic thin-film transistors, solar cells and thermoelectrics. Further extension of this work will focus on the structure modification of this NHC based n-dopant to increase the solubility in commonly used halogenated solvent and modify the doping strength of NHCs.

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Based on N-heterocyclic carbene, a thermally activated n-dopant with excellent air stability and high n-doping efficacy is developed, which can in situ regenerate N-heterocyclic carbene by decarboxylation for the subsequent n-doping. The strategy of thermally activated n-doping not only increases the air stability of n-dopants, but also permits n-dopants to be co-processed with polymer FBDPPV noninteractively before heating in air.