

Solution Processing of a Small Molecule, Subnaphthalocyanine, for Efficient Organic Photovoltaic Cells

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Solution processing of the small molecule subnaphthalocyanine (SubNc) is carried out for the first time to form an electron-donor layer in efficient planar heterojunction organic photovoltaic cells (OPVs). Due to their unique properties, including high solubility, low tendency to aggregate, and strong light absorption in the visible light region, we are able to prepare amorphous SubNc films with high charge-transporting and light-harvesting properties via simple solution casting. By using SubNc as the donor and C₆₀ as the acceptor, we have demonstrated a planar heterojunction OPV with a power conversion efficiency of 1.5%, which represents one of the highest efficiencies for planar heterojunction OPVs based on solution processable small molecules to date. This work clearly shows that solution processing of light-harvesting small molecules has great potential in low-cost thin-film photovoltaic cells. Also SubNc and its derivatives are promising new-generation materials for OPVs.

Introduction

Thin film organic photovoltaics (OPVs) based on small molecules or polymeric materials have attracted significant research attention due to their potential application as low-cost solar energy conversion devices.^{1,2} The power conversion efficiency (PCE) of OPVs has steadily improved over the past decade from about 1% to over 5%, with considerable efforts directed toward the development of new materials and device structures.^{3–8} In the area of device fabrication, there are two primary thin film preparation methods: one is solution processing of soluble materials and the other involves high vacuum vapor deposition of thermally stable molecules. Solution processing is generally believed to be more cost-efficient than vapor deposition, as it does not involve expensive high vacuum systems and is feasible for large-scale roll-to-roll production. Unfortunately, the use of

solution processing in OPVs has so far been limited to soluble conjugated polymers because of their ability to form smooth films upon spin coating. Uniform surface morphology is desirable for better charge separation and charge transport in a solar cell. For example, the combination of poly(3-hexylthiophene) (P3HT), which has good solubility in chloroform and chlorobenzene, with the soluble fullerene derivative [6,6]-phenyl-C61-butyric acid methyl ester (PCBM) represents the state-of-the-art in materials for solution-processed OPVs.^{9,10} To date, there has not been much success in solution processing of small molecules for OPVs despite their advantages over polymeric semiconductors, which include monodispersity, high charge carrier mobility, and relatively simple synthesis.¹¹ To our best knowledge, the state-of-the-art efficiency in solution-processed small molecule PVs has reached only modest values of ca. 1% so far.¹¹ It is generally believed that factors such as limited solubility and tendency to aggregate in most organic solvents are responsible for the difficulty in solution processing of small molecules. For example, copper phthalocyanine (CuPc), a commonly used small molecule donor material, is poorly soluble in most organic solvents; thus, thin films can only be prepared via vapor deposition.¹ In addition, crystallization of solution-cast small molecules on a substrate often results in poor film formation with polycrystalline domains, which

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are characterized by unfavorable grain connectivity and suboptimal crystalline ordering.¹¹

Herein, we demonstrate efficient organic photovoltaic cells based on a solution-processed electron-donor layer using the small molecule subnaphthalocyanine (SubNc). Like its related analogue subphthalocyanine (SubPc), SubNc has a nonplanar pyramid-shaped structure, in which a boron is surrounded by three coupled benzoisindole units to give a 14- π -electron aromatic macrocycle.^{12–14} This unique cone-shaped geometry of SubPc and SubNc contrasts with the flat or nearly flat structure of most phthalocyanines and provides them with distinctive physical properties, such as high solubility and low tendency to aggregate. In addition, their outstanding electronic and optical properties make them of interest for a variety of applications, such as nonlinear optics and photodynamic therapy.^{14–17} The strong absorption in the visible light region with extinction coefficients of ca. $5 \times 10^4 \text{ M}^{-1} \text{ cm}^{-1}$ enables them to be used as effective light-harvesting materials in solar energy conversion applications.¹⁴ Indeed, the concept of artificial photosynthetic systems with SubPcs as light-harvesting donor units has been demonstrated in SubPc- C_{60} dyads.^{18,19} More recently, two research groups have reported efficient organic photovoltaic cells with power conversion efficiency in excess of 2% by using vacuum-deposited SubPc film as the electron donor and fullerene (C_{60}) as the electron acceptor.^{20,21} In this study, we investigated for the first time the use of solution processable SubNc in organic photovoltaic devices. The addition of a ring system to the isindole units increases the solubility of SubNc while reducing its tendency to aggregate and broadening its absorption as compared to SubPc.¹³ These factors enable the solution processing of high-quality SubNc thin films for OPVs.

Experimental Section

Materials. Patterned indium–tin oxide (ITO) coated glass substrates were purchased from Thin Film Devices Inc. Poly(3,4-ethylenedioxythiophene)–poly(styrenesulfonate) (PEDOT:PSS) (Baytron-PH500) was purchased from H. C. Starck. Poly(3-hexylthiophene) (P3HT) used was house-made with a number average molecular weight (M_n) of 35 kDa. Subnaphthalocyanine (SubNc), 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline (BCP) (96%), C_{60}

(99.9%, sublimed), silver shot (1–3 mm, 99.99+%), and chlorobenzene (anhydrous, 99.8%) were purchased from Aldrich. BCP was purified in a home-built thermal gradient sublimator before its use for vapor evaporation.

Devices Fabrication and Measurement. ITO glass substrates were cleaned using the following sequential steps: sonication in soap solution; rinsing with deionized water; boiling in trichloroethylene, acetone, and ethanol for 5 min each; and drying with nitrogen. Finally, the substrates were treated with UV ozone for 10 min. A filtered dispersion of PEDOT:PSS in water (Baytron-PH500) was spun cast at 4000 rpm for 40 s to produce a 32 nm thick layer, followed by baking at 140 °C for 10 min in ambient atmosphere. Solution processing of the films was performed in an inert-atmosphere (argon) glovebox. A solution of SubNc (10 mg/mL in chlorobenzene) and one of P3HT (10 mg/mL in chlorobenzene) were prepared and stirred at 110 °C overnight to ensure complete dissolution. Prior to use in the fabrication of devices, the solutions were passed through a 0.45 μm polytetrafluoroethylene syringe filter. Diluting the 10 mg/mL solution of SubNc with pure chlorobenzene afforded the more dilute solutions (from 2 to 8 mg/mL) used to prepare SubNc films of different thicknesses. Thin films of SubNc and P3HT were prepared by spin-coating their solutions on the PEDOT-precoated ITO substrates at 2000 rpm for 40 s. After solution processing, high vacuum thermal vapor evaporation was applied to deposit the C_{60} , BCP, and silver cathode. The deposition rates for C_{60} , BCP, and silver were controlled at 1.5, 1.5, and 5 $\text{\AA} \text{ s}^{-1}$, respectively. The silver cathode was evaporated through a shadow mask to produce an active area of 0.03 cm^2 . After evaporation, a part of the organic layer was removed to allow contact with the ITO, and then conductive silver paste was painted on the area in order to produce the electrical contact. Thermal annealing was performed on a temperature-controlled hot plate at 120 °C in the glovebox.

All device properties were measured at room temperature in an argon atmosphere under AM 1.5 G solar illumination at 100 mW cm^{-2} (1 sun) using a Thermal-Oriel 300W solar simulator with filter. External quantum efficiency (EQE) values were obtained with a monochromator and calibrated with a silicon photodiode. The Current density–voltage (J – V) characteristics were recorded with a Keithly 236 SMU. Absorption spectra were recorded in a Cary 50 UV–vis spectrometer. Film thickness measurements were performed using a Dektak 150 surface profiler. Topographical images were obtained using a Veeco Multimode V atomic force microscope (AFM).

Results and Discussion

The usefulness of solution-deposited SubNc films in organic photovoltaic cells was evaluated in planar heterojunction devices. Figure 1 shows the chemical structures of the materials, the device structure, and a schematic energy level diagram. The energy levels of each layer are those reported in the literature.^{10,20–23} The device fabrication process involves spin-coating chlorobenzene solutions of the donor materials on top of ITO glass substrates precoated with PEDOT:PSS, followed by vapor deposition of a 325 \AA thick film of C_{60} as acceptor, a 100 \AA thick film of BCP as an exciton blocking layer, and a 1000 \AA thick silver cathode. The detailed device structure and thickness are described as ITO/PEDOT/donor layer/ C_{60} (325 \AA)/BCP (100 \AA)/Ag (1000

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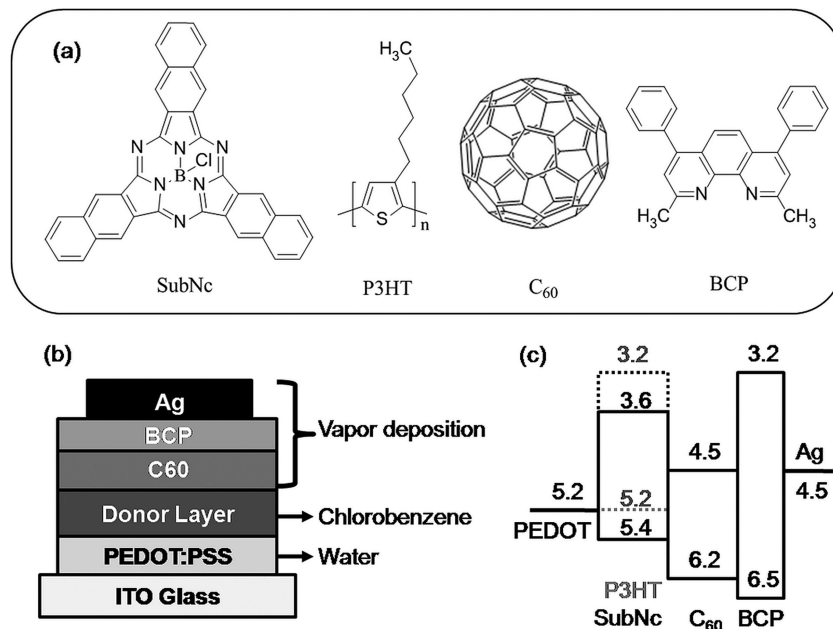


Figure 1. (a) Structures of the materials used in this study. (b) Device architecture and processing methods. (c) Schematic device energy level diagram.

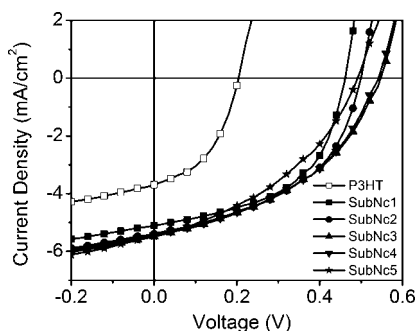


Figure 2. Current density vs voltage (J - V) characteristics of organic photovoltaic cells with the structure ITO/PEDOT/donor layer/C₆₀ (325 Å)/BCP (100 Å)/Ag (1000 Å) under 1 sun AM 1.5 G simulated illumination.

Å), as shown in Figure 1b. The thickness of the SubNc donor layer was controlled by varying the concentration of the solution used for spin-coating and varied from 75 Å (SubNc1) to 320 Å (SubNc5). For valid comparisons, the thicknesses of the C₆₀ and BCP layers were fixed as reported elsewhere.^{20,21} The control sample consisted of a 400 Å thick film of P3HT, a common solution processable polymer donor material.

Figure 2 shows the device performance for the SubNc cells and P3HT control cell under 100 mW cm⁻² AM 1.5 G solar illumination, and Table 1 summarizes the values of each performance parameter for the devices. All the SubNc devices show better performance than the P3HT control device, with higher open-circuit voltages (V_{oc}) of ~ 0.5 V, higher short-circuit current density (J_{sc}) of ~ 5.4 mA cm⁻², and comparable fill factor (FF) of ~ 0.45 . These higher V_{oc} and J_{sc} lead to power conversion efficiencies (η_p) of about 1.25% for the SubNc devices vs 0.3% for the P3HT device. The open-circuit voltage is often considered to be dependent on the energy difference between the lowest unoccupied molecular orbital (LUMO) of the electron acceptor and the highest occupied molecular orbital (HOMO) of the electron donor in planar heterojunction cells.²⁰ Therefore, the in-

creased open-circuit voltage is attributed to the higher energy gap for SubNc/C₆₀ when compared to P3HT/C₆₀, as shown in Figure 1c. For the same reason, the V_{oc} of our SubNc/C₆₀ cell is lower than that of the SubPc/C₆₀ cell, with V_{oc} approaching 1.0 V,^{20,21} as a result of the higher HOMO of SubNc vs SubPc, which is caused by the increased conjugation with the addition of benzo rings.¹³ The J_{sc} is determined by the overlap between the absorption of the active materials and the solar spectrum, the absorption strength, the exciton diffusion length, and the charge transport properties. From absorption and thickness measurements, we calculated that SubNc films have an absorption coefficient of 5×10^4 cm⁻¹, which is similar to that of P3HT. Thus, the spectral overlap of the absorption of the two materials (SubNc and C₆₀) with the solar spectrum may play a larger role in determining the photocurrent of the devices. As shown in Figure 3, SubNc has a much broader absorption than P3HT, peaking at $\lambda_{max} = 688$ vs 556 nm for P3HT, leading to a better overlap with the solar spectrum that has a maximum photon flux around 680–780 nm.²⁴ The extended absorption of the SubNc helps the devices convert more solar illumination to electrons. This is confirmed by comparing the external quantum efficiencies (EQEs) for the SubNc device and the P3HT device. As shown in Figure 4, the SubNc2 cell has a strong photoresponse generated in the region from 650 to 730 nm with EQEs over 10%, which is typical of a planar heterojunction structure. The photocurrent at short wavelengths (400–500 nm) is mainly contributed by C₆₀. Also, due to the extended conjugation and absorption of SubNc, the SubNc/C₆₀ device has a much higher current density (~ 5.4 mA cm⁻²) than the reported SubPc/C₆₀ cell (~ 3.4 mA cm⁻²).²⁰

Unlike many vapor-deposited metal phthalocyanine (MPc)/C₆₀ devices that display a strong performance dependence

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Table 1. Devices Performance under 1 Sun AM 1.5 G Simulated Illumination

cell	donor (Å)	J_{sc} (mA cm ⁻²)	V_{oc} (V)	FF	η_p (%)
P3HT ^a	400	3.57 (3.71)	0.20 (0.25)	0.45 (0.50)	0.33 ± 0.01 (0.47 ± 0.01)
SubNc1	75	5.03	0.46	0.51	1.19 ± 0.04
SubNc2	110	5.39	0.50	0.47	1.25 ± 0.03
SubNc3 ^a	200	5.39 (5.59)	0.55 (0.55)	0.42 (0.49)	1.25 ± 0.03 (1.47 ± 0.02)
SubNc4	285	5.30	0.55	0.43	1.23 ± 0.04
SubNc5	320	5.30	0.49	0.40	1.04 ± 0.02

^a Devices were measured before and after annealing at 120 °C for 40 min; data in parentheses are for annealed devices.

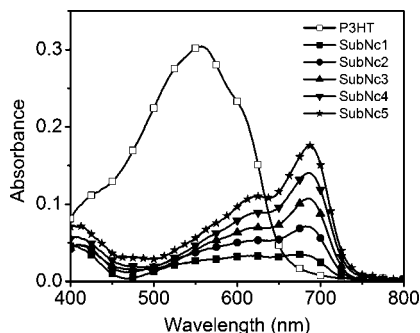


Figure 3. Absorption spectra for the films of P3HT (400 Å) and SubNc (75–320 Å).

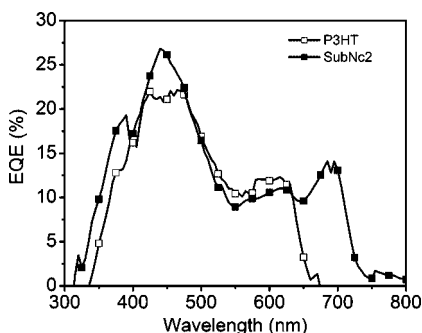


Figure 4. External quantum efficiencies for the P3HT control device and SubNc2 device.

on the thickness of the MPc layer,^{20,25} our devices with solution-processed SubNc film displayed a near thickness-independent behavior up to 300 Å (see Figure 2). This result suggests that the solution-processed SubNc film has a relatively high charge carrier mobility and/or a longer exciton diffusion length (L_{ex}) than many vapor-deposited MPc films.^{20,25} The hole mobility was measured in the device structure of ITO/PEDOT/SubNc (360 Å)/Au based on the field-independent space-charge-limited mobility model. Gold electrode was used to ensure a hole-only device. The measurement was conducted on devices before and after thermal annealing at 120 °C for 40 min, and no significant change in the I – V characteristics was observed. By assuming a relative dielectric constant of 3.9 for SubNc,²¹ the analysis gives a hole mobility of $\sim 2 \times 10^{-5}$ cm² V⁻¹ s⁻¹, which is much higher than that of many other MPcs, such as SnPc with a mobility of $\sim 2 \times 10^{-10}$ cm² V⁻¹ s⁻¹.²⁵ The hole mobility value is also close to the theoretical optimum charge carrier mobility for organic solar cells for minimizing recombination losses as well as ensuring efficient charge

extraction.²⁶ The relatively high mobility of solution-processed small molecule thin film is not surprising when considering the molecular packing and film surface morphology of SubNc. First, the additional aromatic rings increase the extent of conjugation and better overlap between the isoindole units, leading to better charge hopping between molecules in the SubNc film than in the SubPc film.¹⁸ Second, SubNc forms a smooth and continuous film upon solution casting on a substrate, which is beneficial for charge migration without trap sites or shunt currents caused by defects. Atomic force microscopy (AFM) in tapping mode was used to characterize the topology of SubNc thin film on top of PEDOT:PSS-precoated ITO substrate. Figure 5 shows the AFM images of films before and after thermal annealing. It was found that a featureless continuous film was obtained after spin-coating a chlorobenzene solution of SubNc on the substrate. The surface has a root-mean-square (rms) roughness of 16 Å, which is lower than that of vapor-deposited SubPc thin film on ITO substrates,²¹ indicating that high-quality thin films are achievable though solution processing. Thermal annealing at 120 °C for 40 min further reduced the rms roughness to a value of 7 Å. The formation of smooth SubNc films without aggregation can be predicted by comparing the absorption spectra of SubNc in thin films with different thickness and in solution.^{13,17} As shown in Figure 3, the thin film spectra are almost identical to that in solution with little peak shift or change in shape upon changing of thickness, suggesting that molecular aggregation or dimer formation is not prevalent in SubNc films.

It is known that for many organic semiconductor materials exciton diffusion length is primarily determined by the combination of carrier mobility and exciton lifetime. The value of the product (mobility \times lifetime) for high-performance photovoltaic materials with $L_{ex} > 100$ Å is usually of the order of 10^{-10} or 10^{-9} cm² V⁻¹.^{22,27} Given that the hole mobility of SubNc is on the order of 10^{-5} cm² V⁻¹ s⁻¹, the exciton lifetime for SubNc would be expected to be on the order of microseconds. Since SubNc possesses a singlet lifetime of about 3 ns and a triplet lifetime of about 90 μ s in solution,^{14,17} it is likely that the triplet states of SubNc participate in the charge-separation process.²⁸ Such involvement of triplet excitons in SubNc would be expected given its efficient intersystem crossing ($k_{isc} \sim 3 \times 10^8$ s⁻¹) and high triplet quantum yield ($\Phi_T \sim 0.7$).^{14,17} On the basis of these considerations, we believe that SubNc in OPVs behaves very similarly to the typical triplet material PtOEP,

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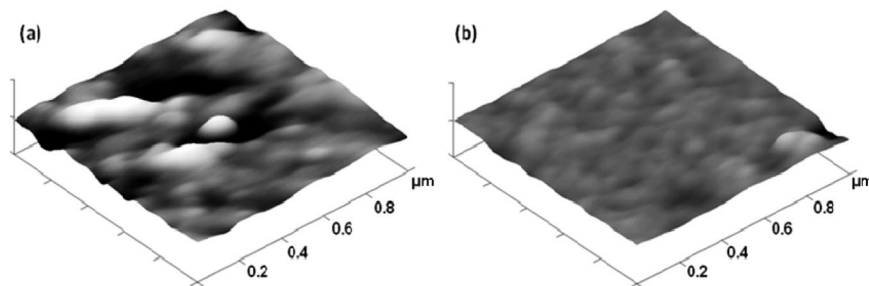


Figure 5. AFM height images of 200 Å thick film of SubNc on top of PEDOT:PSS-precoated ITO substrates (vertical axis is on a scale of 20 nm/division, horizontal axes are 0.2 μm/division): (a) before annealing with rms roughness of 16 Å and (b) after thermal annealing at 120 °C for 40 min with rms roughness of 7 Å.

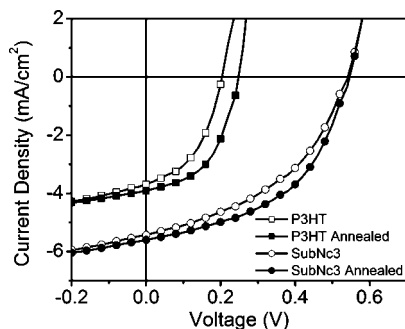


Figure 6. Current density versus voltage (J - V) characteristics of the P3HT control device and SubNc3 device under 1 sun AM 1.5 G simulated illumination, before and after thermal annealing at 120 °C for 40 min.

which has an almost identical hole mobility of about $10^{-5} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$, a triplet lifetime of 91 μs in a doped thin film, and an exciton diffusion length of around 300 Å.²² The fundamental study of triplet excitons behavior in OPVs based on SubNc and its related materials is in process.

We further investigated the effect of thermal annealing on device performance. Thermal treatment has been shown to be effective in improving device performance by reducing the series resistance in many OPVs.^{5,22} In our study, the devices were treated at 120 °C in an argon-filled glovebox for 40 min. Figure 6 shows the device performance for the SubNc3 cell and the P3HT control cell before and after thermal treatment. It is found that thermal annealing significantly improved the P3HT device efficiency from 0.34% to 0.48%, with a concomitant increase in all the important device characteristics (V_{oc} , J_{sc} , and FF). This improvement can be ascribed to the enhanced charge carrier mobility of P3HT thin film caused by thermally induced crystallization.²⁹ Although not as pronounced as in the P3HT control device or in other small molecular OPVs,^{5,22} an improvement in performance is also observed for the SubNc cell upon heat treatment. The increase in PCE value from 1.28% to 1.49%

upon annealing is mainly due to the increase in fill factor (FF) from 0.43 to 0.49. Since there was almost no change in film morphology or carrier mobility for the SubNc layer upon thermal annealing as described above, the slight improvement is most probably caused by the increase of donor/acceptor interface area as a result of the interdiffusion of SubNc and C_{60} layers during thermal annealing.

Conclusion

In summary, we have demonstrated efficient organic photovoltaic cells based on the solution processable small molecule subphthalocyanine (SubNc). The unique structural and photophysical properties of SubNc, including high solubility, low tendency to aggregate, and strong light absorption, enable the solution processing of thin films with high light-absorbing and charge-transporting properties. By using SubNc as the donor and C_{60} as the acceptor, a power conversion efficiency of 1.5% was achieved in a simple planar heterojunction structure with an open circuit voltage of 0.55 V, a short circuit current density of 5.6 mA cm^{-2} , and a fill factor of 0.49, which represents one of the highest performing devices based on solution processable small molecules. The participation of triplet excitons of SubNc is likely a factor contributing to the high device performance. Our study clearly shows that the use of solution-processed small molecules is promising in the search for low-cost efficient organic photovoltaic devices and that SubNc and its derivatives are promising as new-generation materials for OPVs. We are currently investigating fully solution-processed OPVs by using SubNc derivatives as the active materials, which involves bulk heterojunction structure devices and bilayer devices applying cross-linking strategy.

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