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# Synthesis of 3*H*-benzo[e]indoline and Its Application to Small-Molecule Organic Solar Cells

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**Abstract:** A squaraine-based small molecule (**USQ-BI**) bearing 3*H*-benzo[e]indoline was synthesized as an electron donor, and the corresponding organic solar cells show power conversion efficiency of 5.35% with an excellent short circuit current over 15 mA/cm<sup>2</sup>. At the same time, the hole mobility of **USQ-BI** was about 5 times (9.57×10<sup>-5</sup> vs. 2.00×10<sup>-5</sup> cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>) higher than that of indoline-based squaraine.

Indole is one of the most popular motifs in natural bioactive products, commercial drugs, and other functional materials.<sup>[1]</sup> Indole and its derivatives can be synthesized through a variety of methods, such as Leimgruber-Batcho indole synthesis, Fischer indole synthesis, and the latter is the most widely adopted approaches.<sup>[1a, 2]</sup> Nowadays, a lot of indole families were synthesized, and many of them have been utilized to construct organic semiconductors.<sup>[3]</sup>

Indoline, as an electron-richer indole derivative, has received increasing attention in the field of dye-sensitized solar cells and organic solar cells,<sup>[3a]</sup> and most of their derivatives have achieved high performance.<sup>[3a, 4]</sup> Here, to promote more effective intermolecular  $\pi$ - $\pi$  stacking, we designed and synthesized 3*H*-benzo[e]indoline, a novel benzene-ring-fused indoline, to enhance the charge transport for the organic semiconductors.<sup>[5]</sup>

Squaraine dyes have drawn much attention for small molecule organic solar cell (SMOSCs), due to their intense absorption in the visible-NIR region, their aood photochemical/photophysical stability, and. most importantly, the facile synthetic access without involving the highly toxic reagents, which are very important for the commercial application. <sup>[3c, 4b, 6]</sup> In 2014, we designed and synthesized unsymmetrical squaraine (USQ)-based small molecule using indoline (ASQ-5) as an electron donor for organic solar cell.[3b] And the corresponding photovoltaic device showed the short circuit current  $(J_{sc})$  of 11.03 mA/cm<sup>2,[3b]</sup> then boosted the PCE of USQ-based bulk heterojunction (BHJ) SMOSCs to 4.29%. Although indolinebased squaraine dyes exhibited better photovoltaic performance than other squaraine dyes with dialkylamino, 9carbazyl and diphenylamino, [3b, 7] its limited hole-transport properties seemed to represent a bottleneck as suggested by the generally observed low FF.<sup>[8]</sup>

In this contribution, we designed and synthesized a novel squaraine dye (**USQ-BI**) with the 3*H*-benzo[e]indoline to enhance its charge transport (Figure 1).<sup>[9]</sup> In comparison with non-benzene-ring-fused **ASQ-5**, **USQ-BI** shows about 5 times higher hole-mobility ( $9.57 \times 10^{-5}$  vs.  $2.00 \times 10^{-5}$  cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>) in pristine film, and the corresponding photovoltaic devices exhibited a higher performance of 5.35%, with a  $J_{sc}$  over 15 mA/cm<sup>2</sup> which is the highest value for the squaraine-based OSCs.<sup>[6a]</sup>

The synthetic routes to the 3*H*-benzo[e]indoline and **USQ-BI** are illustrated in Figure 1. All the synthetic and characterization details could be found in supporting

[a] Y. Chen, J. Wu, L. Yang, B. Hu, Dr. D. Yang, Prof. Z. Lu, Dr. Y. Huang Key Laboratory of Green Chemistry and Technology (Ministry of Education) College of Chemistry, Sichuan University, Chengdu 610064 (P. R. China) information. 3*H*-benzo[e]indole was synthesized through Fischer Indole Synthesis, then it was converted into 3*H*benzo[e]indoline (5). The benzo[e]indole semi-squarylium compound 8 condensed with 3*H*-benzo[e]indoline-based compound 7 obtain the **USQ-BI**. **USQ-BI** possess a good solubility in chloroform (~30 mg mL<sup>-1</sup>), suggesting a highquality film could be expected through solution-processing.

The single crystal of **USQ-BI** was obtained from the dichloromethane/methanol.<sup>[10]</sup> From the crystal structure (Figure 2), the OH···O bond lengths were measured to be 1.764 and 1.676 Å, indicating the existence of strong hydrogen bonding interactions. As a result, the squarate cores and the phenyl moieties shown quasi-coplanar conformations with a dihedral angle of 4.39°, then the whole molecule also exhibits a good planar structure. According to the packing diagrams of **USQ-BI**, the distance between two neighbouring molecules is 3.63 Å, indicating the presence of significant  $\pi$ - $\pi$  interaction, which may facilitate the charge carrier transportation.<sup>[11]</sup>



Figure 1. The synthetic route for the 3*H*-benzo[e]indole and relative 3*H*-benzo[e]indoline.



Figure 2. The crystal structure(a) and stacking model of the USQ-BI(b).

The optical absorption spectra of the **USQ-BI** in solution and as thin films are shown in Figure 3, and all optical data are summarized in the Table 1. Compared to the **ASQ-5**, **USQ-BI** exhibits slightly lower molar extinction coefficient ( $2.00 \times 10^5 vs$ .  $2.34 \times 10^5 L mol^{-1} cm^{-1}$ ) but red-shifted absorption (691 nm vs. 678 nm), larger width at half maximum (FWHM) (47 nm vs. 39 nm) in dilute solution, due to the extended conjunction from the 3*H*-benzo[e]indoline. When going from solution to thin-film state, the absorption spectrum is red-shifted and displays an intense and broad absorption band in the range 550–850 nm with a FWHM of 174 nm (166 nm for **ASQ-5**), suggesting that the **USQ-BI** shows stronger  $\pi$ -  $\pi$  stacking than that of **ASQ-5**. Combination of the molar extinction coefficient and FWHM, the blend films of **USQ-BI** and **ASQ-5** show comparable absorption (Figure S2).

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From the low energy absorption onset of the thin-film states, the optical bandgap is estimated to be 1.39 eV, which is smaller than that of the **ASQ-5** (1.43 eV).<sup>[12]</sup>

According to the cyclic voltammetry (CV) measurements (Figure 3c), the highest occupied molecular orbital (HOMO) levels of **USQ-BI** are estimated to be at -5.05 eV which is slightly higher than that of **ASQ-5** (-5.09 eV). The higher HOMO level which could be the extended conjugation, and it consistent with the optical results.

To evaluate **USQ-BI** photovoltaic properties, BHJ-OSC cells were fabricated using [6,6]-phenylC<sub>71</sub>butyric acid methyl ester (PC<sub>71</sub>BM) as the electron acceptor material with a conventional device structure (ITO/MoO<sub>3</sub> (8 nm)/USQs: PC<sub>71</sub>BM (~80 nm)/ LiF (0.8 nm)/Al (100 nm)). Photovoltaic data of these devices are summarized in Table 2 and Table S2, and representative current density-voltage (*J*-*V*) and external quantum efficiency-wavelength (EQE) curves are shown in Figure 4 and Figure S3.

Compared to the **ASQ-5**-based device with a D/A ratio of 1:5, the **USQ-BI**-based devices with the same ratio exhibit higher  $J_{\rm sc}$  (12.02 vs 10.29 mA/cm<sup>2</sup>) and higher FF (0.52 vs 0.45) with open-circuit voltage ( $V_{\rm oc}$ ) of 0.74 V (0.82 V for **ASQ-5**- based device).<sup>[3b]</sup>



Figure 3. Molecule structure(a), Optical absorption spectra in solution and as thin films (b) as well as the cyclic voltammograms (c) of the USQ-BI and ASQ-5.

Table 1 Optical, electrochemical and electric properties of USQ-BI and ASQ-5.

	<u>λ<sub>max</sub> (nm)</u>		FWHM (i	nm)	E	Earset	HOMO	LUMO
Compd	Solution(ɛ10 <sup>5</sup> )	Film	Solution	Film	(eV)	(V)	(eV)	eV
USQ-BI	691 (2.00) <sup>a</sup>	737 <sup>b</sup>	47	174	1.39	0.25 °	-5.05	-3.66 <sup>d</sup>
ASQ-5 <sup>e</sup>	678 (2.34)	725	39	166	1.43	0.29	-5.09	-3.66
*Measured in	dilute chloroform s	olution (2	.00×10 <sup>-6</sup> M); <sup>4</sup>	Measur	red in thin f	ilm state; °	Derived from	CV

Since the  $V_{oc}$  is highly correlated with the HOMO level of the donor materials, the lower  $V_{oc}$  of **USQ-BI**-based devices could be ascribed to the lower HOMO level of **USQ-BI** (-5.05 vs. -5.09 eV) due to the extended conjugation. Furthermore, the devices based on **USQ-BI** show a much higher fill factor (FF) (0.52 vs. 0.45), and it's one of highest value for squaraine-based device. Actually, the squaraine-based OSCs usually shown a relative lower FF than other high-performance materials which is the main limitation for the squaraine-based device.<sup>[6a]</sup> Since the **USQ-BI**-based device shown a higher  $J_{sc}$  and FF, the **USQ-BI**-based devices show the PCE of 4.68%, which is much higher than **ASQ-5**-based device (3.80%) under a D/A ratio of 1:5 without any treatments.

The PCE of the device is highly correlated with the charge mobility and morphology.<sup>[13]</sup> We further study the charge mobility through space-charge-limited current (SCLC) method and morphology through atomic force microscopy (AFM). According to the experimental and calculation results (Figure S4), the hole mobility( $\mu_h$ ) of the as-cast **USQ-BI** neat film is 9.57 × 10<sup>-5</sup> cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> which is about 5 times higher than that of **ASQ-5** (2.00 × 10<sup>-5</sup> cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>), demonstrating the simple benzene-ring-fused strategy is very effective for indoline skeleton. And the hole mobility of **USQ-BI**: PC<sub>71</sub>BM blending film with a ratio of 1:5 is 5.8 × 10<sup>-5</sup> cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>, which is also much higher than that of **ASQ-5** 

 $(1.71 \times 10^{-6} \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1})$ . The difference in hole mobility observed for **USQ-BI** and **ASQ-5** can most likely be ascribed to the extending conjugation using a much larger planner structure, which can result in higher carrier mobility.<sup>[5a]</sup> The surface morphologies of the blended films of **USQ-BI** and **ASQ-5** have been investigated by AFM (Figure S5), they show similar morphologies and quite smooth surfaces with root-mean-square (RMS) roughness of 0.27-0.32 nm, respectively. Hence, the higher PCE of the **USQ-BI**-based SMOSCs which can be ascribed to enhanced hole mobility.

Table 2 Photovoltaic performance of BHJ-OSCs based on  $\ensuremath{\text{USQ-BI}}$  and  $\ensuremath{\text{ASQ-5}}$  .

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Donor	Device	Voc	J <sub>sc</sub>	FF	PCE
	(D: A)	$(\vee)$	(mA/cm <sup>2</sup> )		(%)
USQ-BI	1:5	0.74±0.01	12.02±0.45	0.52±0.01	4.68±0.32
USQ-BI	1:5ª	0.73±0.01	13.19±0.20	0.52±0.01	5.04±0.13
USQ-BI	1:3	0.72±0.01	12.67±0.12	0.44±0.01	4.02±0.22
USQ-BI	1:3ª	0.73±0.01	15.64±0.38	0.47±0.01	5.35±0.25
ASQ-5 <sup>b</sup>	1:5	0.82	10.29	0.45	3.80
ASQ-5 <sup>b</sup>	1:5ª	0.81	11.03	0.48	4.29

<sup>a</sup>The active layer was thermal-annealed at 70 °C for 20 min. Data obtained from the average of 8 individual devices.<sup>b</sup> Data from reference 3b.



**Figure 4.** Representative *J-V* characteristics of the OSC devices (a D: A=1:5, b D: A=1:3).



**Figure 5.** a) Measured  $J_{sc}$  against light intensity on a logarithmic scale, b) Measured  $V_{oc}$  against light intensity.

In order to fully explore the potential of novel indoline-based semiconductors for the organic functional materials, the OSCs were fabricated in different D/A ratios with/without thermal annealing (TA). As shown in Table 2 and S2, the devices with a 1:3 ratio show the highest PCE of 4.68% among all the devices without TA. Upon TA, the devices exhibit 5-15% increase for the same ratio except 1:3, which are the similar case for most of SQ-based OSCs, including **ASQ-5**.<sup>5</sup> For the ratio of 1:3, the PCE of 5.35% was achieved with after thermal annealed at 70 °C for 20 min (Table 2), 33% higher than that of the as-cast devices, which is originated from the significantly increasing of  $J_{sc}$  and a slightly increasing of FF and  $V_{oc}$ . Please note that the  $J_{sc}$  is high to 15.64 mA/cm<sup>2</sup>, to the best of our knowledge, which is the highest value among SQ-based OPV.

To gain further insights of the effects of thermal annealing on the blend films with the ratio of 1:3 and 1:5, the charge mobility, microstructure, and morphologies were investigated by SCLC devices, GIXRD, AFM and TEM. After thermal annealing, the hole mobility slightly increased (8.23 ×  $10^{-5}$  vs. 7.09 ×  $10^{-5}$  cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup>), with the similar variation of the ratio of 1:5 (4.66×  $10^{-5}$  vs. 4.05 ×

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 $10^{\text{-5}}\ \text{cm}^2\ \text{V}^{\text{-1}}\ \text{s}^{\text{-1}}).$  The measurements of GIXRD, AFM and TEM all exhibit the similar cases (Figures S5-7).

To eliminate the possibility of experiment errors, two more batches of devices in the ratio of 1:3 with/without TA were reproduced carefully, and all the data show the same results, even including the slightly increase of  $V_{\text{oc}}$  and FF. And these increases may be caused by weaker charge recombination.<sup>14</sup> Then, light intensity (I) dependent J-V analysis of the OSCs were performed (Figure 5) to investigate the charge recombination kinetics. The relationship between  $J_{SC}$  and I is defined as  $J_{SC} \propto I^S$ , where the S value close to the 1, which means it shows a weak bimolecular recombination.<sup>14</sup> As shown in the Figure 5, both of them show the same slope of 0.96, suggesting that TA treatment has no effects on the bimolecular recombination. The relationship between  $V_{OC}$  and I should be  $V_{OC} = n(k_BT/e) \ln(I) + C$ , where n is the ideality constant,  $k_B$  the Boltzmann constant, T temperature, e elementary charge, and C a constant.<sup>14</sup> When n = 1, there exist only bimolecular recombination; when n>1, there exist the mixture of bimolecular recombination and monomolecular recombination. Interestingly, the value of n was decreased after TA from 1.37 to 1.16, indicating TA treatment can significantly decrease the monomolecular recombination. And this decrease can lead to the increase of  $J_{sc}$ , FF and  $V_{oc}$ .

In addition, the blend film with 1:3 ratio exhibit much stronger absorption in comparison the film with 1:5 ratio (Figure S2). Therefore, the much increased  $J_{sc}$  after TA could be rationally ascribed to the stronger absorption, higher charge mobility, and weaker monomolecular recombination.

In conclusion, a novel unsymmetrical squaraine with 3Hbenzo[e]indoline was designed, synthesized, and characterized. The hole mobility of USQ-BI was about 5 times compared to with that of indoline-based squaraine (ASQ-5) in pristine film. The solution-processed small-molecule BHJ OSCs fabricated with USQ-BI/ PC71BM exhibited a high PCE of 5.35% with the excellent  $J_{sc}$  over 15 mA/cm<sup>2</sup>, which is one of the highest value for the squaraine-based OSCs. And higher PCE could be expected with electron deficient substituents (-F, -CN, et al.) on the indoline. These preliminary studies demonstrate that the simple benzene-ring-fused strategy is very effective for indoline skeleton to increase the hole mobility and the photovoltaic performance. At the same time, these results demonstrate that 3H-benzo[e]indoline is a promising constructing-unit for OSCs, and it can be extended to construct other high-performance organic semiconductors.

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#### **Conflict of interest**

The authors declare no conflict of interest.

**Keywords:** Indoline • Squaraine • Organic Solar Cells • Charge Mobility

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Layout 1:

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A new indoline derivative, 3Hbenzo[e]indoline, was synthesized through Fischer indole synthesis, and its applications to the smallmolecule photovoltaics have been explored with a promising short circuit current over 15 mA/cm<sup>2</sup>.



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