Organic Solar Cells

A Halogenation Strategy for over 12% Efficiency Nonfullerene Organic Solar Cells

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Three acceptor–donor–acceptor type nonfullerene acceptors (NFAs), namely, F–F, F–Cl, and F–Br, are designed and synthesized through a halogenation strategy on one successful nonfullerene acceptor FDICTF (F–H). The three molecules show red-shifted absorptions, increased crystallinities, and higher charge mobilities compared with the F–H. After blending with donor polymer PBDB-T, the F–F-, F–Cl-, and F–Br-based devices exhibit power conversion efficiencies (PCEs) of 10.85%, 11.47%, and 12.05%, respectively, which are higher than that of F–H with PCE of 9.59%. These results indicate that manipulating the absorption range, crystallinity and mobilities of NFAs by introducing different halogen atoms is an effective way to achieve high photovoltaic performance, which will offer valuable insight for the designing of high-efficiency organic solar cells.

Owing to the deeper understanding of molecular design, device optimization, and operating mechanism, organic solar cells (OSCs) have made great progress in the past decade. So far, power conversion efficiencies (PCEs) over 11% have been achieved for the devices based on fullerene acceptors (FAs),^[1-8] and PCEs over 13% have been realized for devices based on nonfullerene acceptors (NFAs).^[9-11] NFA design is still the major driving force to pursue high performance of OSCs. In the past decade, especially in recent three years, many great small molecule-based NFAs with different chemical structures, such as with acceptor-donor-acceptor (A-D-A) backbone architectures^[9,12-16] and naphthalene diimide (NDI), perylene diimide (PDI), diketopyrrolopyrrole (DPP) based NFAs,^[17-27] etc., have been designed and evaluated. To date, NFAs with A-D-A structure have earned great success and PCEs over 13% have been achieved.^[9-11] The design strategies of A-D-A-type NFAs are focused on two aspects: the donor core units and the

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acceptor end-groups,^[12,13,15,28–36] aiming to tune the energy levels, absorption, and even packing modes in the active layers. In contrast to the various core units for the A–D–A-type NFAs, only a few endgroups such as 2-(2,3-dihydro-3-oxo-1Hinden-1-ylidene)propanedinitrile (INCN) and rhodanine and their derivatives have been reported.^[11,32,34–37] The optimization of end-groups, especially delicate modification of the successful end-group INCN has been proved to exert great influence not only on the energy levels, but also on the intra- and intermolecular packing states of the NFAs.^[9,37–39]

Halogens have been widely used in the organic semiconductors.^[40,41] The roles of

fluorine (F) atom in OSCs materials have been well studied.^[42–45] It has been found that the electronegativity of the substituted sites could be improved and the bathochromic absorption would be obtained after introducing the F atom. In addition, the possible internal F–S and F–H noncovalent interaction might be beneficial to improve the intermolecular packing and thus promoting charge transport properties.^[9,12,43,45] Also, few studies have addressed on the materials incorporating the Cl and Br in bulk-heterojunction OSCs. Just recently, Li and co-workers have reported a series of halogenation NFAs based on 3,9-bis(2-meth-ylene-(3-(1,1-dicyanomethylene)-indanone)-5,5,11,11-tetrakis(4-hexylphenyl)-dithieno[2,3-d:2',3'-d']-s-indaceno[1,2-b:5,6-b']-dithiophene (ITIC) and PCE over 9% has been achieved.^[37]

Recently, our group has reported an NFA named 2,9-bis-(2methylene(3(1,1dicyanomethylene)indanone))7,12-dihydro-4,4,7,7,12,12-hexaoctyl-4H-cyclopenta[2",1":5,6;3",4":5',6']diindeno[1,2-b:1',2'-b']dithiophene (FDICTF) (F-H) (Figure 1a).^[15] When blended with the wide bandgap polymer poly[(2,6-(4,8bis(5-(2-ethylhexyl)thiophen-2-yl)-benzo[1,2-b:4,5-b'] dithiophene))-alt-(5,5-(1',3'-di-2-thienyl-5',7'-bis(2-ethylhexyl) benzo[1',2'-c:4',5'-c']dithiophene-4,8-dione))] (PBDB-T), its based device demonstrated a PCE of 10.06%. Taking account of the unique advantages of halogenated molecules, in this work, three NFAs, namely, F-F, F-Cl, and F-Br (Figure 1a), using the same conjugated core of F-H and introducing F, Cl, and Br onto the end-capping group INCN, respectively, have been designed and synthesized. Compared with F-H, the lowest unoccupied molecular orbital (LUMO) levels of the three molecules decreased with different degrees, owing to the electron-withdrawing difference of the halogen atoms. When blended with PBDB-T as the donor material, the F-F-, F-Cl-, and F-Br-based

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Figure 1. a) Chemical structures of F–H, F–F, F–Cl, and F–Br. b) Energy-level diagrams of PBDB-T, F–H, F–F, F–Cl, and F–Br using the UPS method. c) Absorption spectra of F–H, F–F, F–Cl, and F–Br in chloroform solutions, d) in solid films.

optimized devices exhibited PCEs of 10.85%, 11.47%, and 12.05%, respectively. The significantly improved performances of their devices demonstrated that the halogenation is another effective strategy for designing high-efficiency NFAs.

The three molecules, F-F, F-Cl, and F-Br, were synthesized between the dialdehyde intermediate and corresponding ending groups by the Knoevenagel condensation. The detailed procedures were described in the supporting information (SI). Since the ending groups of F–F. F–Cl. and F–Br are composed of two isomers, which are hard to be separated, the three materials are also used as their corresponding isomer mixtures. The three molecules were fully characterized by ¹H NMR, ¹³C NMR, and high resolution mass spectrum. They exhibit good solubility in the comment solvents, such as chloroform, dichloromethane. and ortho-dichlorobenzene. Thermogravimetric analysis (Figure S2, Supporting Information) was used to investigate their thermal stability under nitrogen atmosphere. The weight loss was lower than 5% until the temperature reached 320 °C, indicating that the thermal stabilities of these three molecules are adequate to be applied in organic photovoltaics (OPV) device.

The ultraviolet-visible (UV–vis) absorptions of F–F, F–Cl, and F–Br in chloroform solution and thin film were measured,

and the corresponding data with those of F-H for comparison are summarized in Table 1. As shown in Figure 1c, in chloroform solutions, all of them exhibit strong absorption in the range of 550-700 nm, and the maximum absorption peaks of F-F, F-Cl, and F-Br are 674, 678, and 679 nm, respectively. In the film states (See Figure 1d), the maximum absorption peaks of F-F, F-Cl. and F-Br are red-shifted by 29, 33, and 41 nm, respectively, compared with their solution states. It is worthy to note that the absorption peaks of the three molecules whether in solution or on solid are red-shifted compared to that of F-H. In their dilute chloroform solutions, F-H, F-F, F-Cl, and F-Br have similar high maximum molecular extinction coefficients (Figure S3, Supporting Information). But as shown in Table 1, the extinction coefficients of halogenated molecules (F-F, F-Cl. and F-Br) in their solid films are significantly higher than that of F-H, which might be ascribed to the hyperchromic effect of halogen atoms and better packing, as indicated in the grazing incidence wide angle X-ray scattering (GIWAXS) results shown below. This is also consistent with the results of optical band gaps (E_{σ}^{opt}), estimated from the absorption edge of F–F, F–Cl, and F-Br, which are 1.59, 1.58, and 1.56 eV, respectively, and slightly narrower than 1.63 eV of F-H material.

Table 1.	The optical	data and	molecular	energy	levels o	f F–H,	F–F, F–Cl	, and F–Br.
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Molecules	$\lambda_{\max}^{soln.}$ [nm]	$\lambda_{\max}^{\text{fil.}}$ [nm]	$\lambda_{edge}^{fil.}$ [nm]	$E_{\rm g}^{\rm opt.} [\rm eV]^{a)}$	HOMO [eV]	LUMO [eV]	[10 ² nm ⁻¹] ^{b)}
F–H	665	689	760	1.63	-5.42	-3.79	0.78
F-F	674	703	780	1.59	-5.72	-4.13	0.97
F–Cl	678	711	786	1.58	-5.62	-4.04	0.93
F–Br	679	720	793	1.56	-5.62	-4.06	0.98

 $^{a)}E_{g}^{opt} = 1240/\lambda_{edge}$ (eV), $^{b)}$ The values of ε were measured for the film of the materials prepared from their solutions.

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Acceptor	V _{oc} [V]	J _{sc} [mA cm ⁻²]	FF	PCE _{max} (PCE _{ave}) (%) ^{a)}	J_{calc} . [mA cm ⁻²] ^{b)}
F–H	$\textbf{0.94}\pm\textbf{0.01}$	15.02 ± 0.26	$\textbf{0.67} \pm \textbf{0.01}$	9.59 (9.38)	14.39
F-F	$\textbf{0.88} \pm \textbf{0.01}$	17.36 ± 0.33	0.71 ± 0.01	10.85 (10.67)	16.52
F–Cl	$\textbf{0.87}\pm\textbf{0.01}$	17.61 ± 0.13	$\textbf{0.75} \pm \textbf{0.01}$	11.47 (11.26)	16.81
F–Br	$\textbf{0.87}\pm\textbf{0.01}$	18.22 ± 0.16	$\textbf{0.76} \pm \textbf{0.01}$	12.05 (11.88)	17.37

Table 2. Device performance parameters of OSC based on PBDB-T and four acceptors measured at simulated 100 mW cm⁻² AM 1.5G illumination.

 $^{a)}\mbox{Average}$ values from 20 devices, $^{b)}\mbox{J}_{sc}$ integrated from the EQE spectrum.

The electrochemical properties of the three molecules were investigated by cyclic voltammetry (CV). Using the ferrocene/ferrocenium (Fc/Fc⁺) redox couple (4.8 eV below the vacuum level) to internally calibrate the potentials. The highest occupied molecular orbital (HOMO) and the LUMO energy levels are calculated from the onset oxidation and reduction potentials of the redox curves. The HOMO/LUMO levels (Figure S9, Supporting Information) of F-F, F-Cl, and F-Br are -5.45/-3.77, -5.46/-3.75, and -5.47/-3.78 eV, respectively. Due to the electron-withdrawing effect of halogen atoms, the LUMO levels decreased in comparison with that of the F-H. We also used ultraviolet photoemission spectroscopy (UPS) to investigate the energy levels in their solid state, as shown in Figure S5 in the Supporting Information, and the HOMO levels could be calculated using the incident photon energy, $h\nu$ = 21.22 eV, E_{cutoff} , and E_{onset} . The LUMO levels were calculated with the HOMO levels and E_{g}^{opt} . The HOMO/LUMO levels of F-H, F-F, F-Cl, and F-Br are -5.42/-3.79, -5.72/-4.13, -5.62/-4.04, and -5.62/-4.06 eV, respectively. Although there is little difference between the energy levels determined from the CV and UPS measurements, the trends are similar. The HOMO and LUMO levels of PBDB-T were also determined to be -5.18 and -3.38, respectively, using the UPS method. For the PBDB-T:F-F/F-Cl/F–Br systems, the ΔE_{HOMO} and ΔE_{LUMO} are larger than those of F-H system, which are beneficial for the excitons dissociation and transfer between the donor and acceptors. The energy levels of these molecules using the UPS method are shown in Table 1 and Figure 1b. In order to further investigate the molecular geometries and electronic properties of F-F, F-Cl, and F-Br, quantum chemistry calculation by density functional theory at B3LYP/6-31G* level was conducted. All the side-chains were replaced by methyl groups for simplification, and the optimized geometry of the three molecules and F-H are shown in Figure S1 in the Supporting Information. The result indicates that the backbones of the three molecules have nearly planar configurations, which are nearly the same as that of F-H. For F-F, F-Cl, and F-Br, the electron density of HOMO is mainly distributed along the conjugated cores, while the LUMO is delocalized over the entire molecules.

Photoluminescence (PL) spectroscopy was used to evaluate the molecular exciton quenching efficiencies in the blend films, and the results were shown in Figure S6 in the Supporting Information. Similar to F–H,^[15] the pure F–F, F–Cl, and F–Br films had strong PL intensities in the regions of 700–850 nm. When blended with PBDB-T, all the blend films showed obvious fluorescence quenching, suggesting that effectively photo-induced charge transfer occurred between the donor and acceptors.

In order to assess the photovoltaic properties of these acceptors, OPV devices were fabricated with a conventional device architecture of ITO/PEDOT:PSS/PBDB-T:acceptors/PDINO/Al, where PDINO is an efficient cathode interlayer.^[46] In addition, we used the same batch polymer donor (PBDB-T) and device architecture to revaluate the photovoltaic performance of the F-H, and similar results were obtained compared to our previous work (Table 2).^[15] The optimized D/A weight ratios for PBDB-T:F-F/F-Cl/F-Br are 1:1.2, 1:1, and 1:1, respectively, with the optimized thickness about 150 nm. The photovoltaic devices based on PBDB-T:F-F/F-Cl/F-Br casted from chlorobenzene with 0.3%/0.1%/0.3% 1,8-Diiodooctane (DIO) showed best PCEs of 10.85%, 11.47%, and 12.05%, respectively. The detailed device parameters for device optimization were described in Tables S1-S3 in the Supporting Information. Due to their similar LUMO levels and gradually red-shifted UV-vis spectra absorption from F–F to F–Br, these three NFA-based devices showed similar $V_{\rm oc}$ of 0.88, 0.87, and 0.87 V, which are lower than that of F-H-based device. It is worthy to note that F-Cl- and F-Br-based devices gave fill factors (FFs) with values over 75%, which are among the NFA OSCs with the highest FFs. The typical current density versus voltage (I-V) characteristics are illustrated in Figure 2a, and the corresponding photovoltaic parameters with those of F-H for comparison are summarized in Table 2. External quantum efficiency (EQE) curves are shown in Figure 2b to confirm the J_{sc} of the OSCs. For the optimized devices based on these three molecules, similar with F-H, but a general higher and broader photo-to-current response in the range of 300-780 nm were achieved, and the PBDB-T:F-Cl-/F-Br-based devices exhibited a broader photon response range than PBDB-T:F-F based device (Figure 2b). But, in the range of 400-450 and 650-720 nm, F–Br-based devices showed higher responses than that of F–Cl, and a maximum value of ${\approx}80\%$ was recorded at 560 nm. The integrated current density was 16.52/16.81/17.37 mA cm⁻² for the PBDB-T:F-F/Cl/Br based devices, which agrees well with the I-V measurements. Furthermore, the devices were stored in glove box without encapsulation to study their stability. As shown in Figure S7 in the Supporting Information, after 10 d, the PCEs of F-H/F-F/F-Cl/F-Br based devices remain 68%/78%/73%/71% of their initial values, respectively.

The relationship between the photocurrent density ($J_{\rm ph}$) and effective applied voltage ($V_{\rm eff}$) was measured to further investigate the high performance and FFs of the devices. $J_{\rm ph}$ is defined as $J_{\rm ph} = J_{\rm Light} - J_{\rm D}$, where $J_{\rm Light}$ and $J_{\rm D}$ are the photocurrent densities under light illumination and in the dark, respectively. $V_{\rm eff} = V_0 - V_a$, where V_0 is the applied voltage where $J_{\rm ph}$ is zero and V_a is the applied voltage.^[47] As we all know, $V_{\rm eff}$ are related to the internal electric field in the devices, which can influence the exciton dissociation and charge collection properties. From the Figure 2c, we can find that the $J_{\rm ph}$ increased with the raise of $V_{\rm eff}$ and all the $J_{\rm ph}$ values could reach saturation ($J_{\rm sat}$) when



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Figure 2. a) Current density–voltage (*J*–V) curves of the devices based on PBDB-T and F–X (H, F, Cl, Br) under the illumination of AM 1.5G (100 mW cm⁻²). b) EQE spectra of the devices. c) J_{ph} versus V_{eff} and d) light-intensity (*P*) dependence of J_{sc} of the devices.

 $V_{\rm eff}$ arrived at ≈2 V. To further compare the charge dissociation and charge collection probability (*P*(*E*, *T*)) in the three devices, the value of ($J_{\rm ph}/J_{\rm sat}$) was adopted.^[48] Under their short-circuit conditions, compared with 91% for the F–H-based devices, the $J_{\rm ph}/J_{\rm sat}$ ratios were 91%, 93%, and 94% for the F–F-, F–Cl-, and F–Br-based devices, suggesting high exciton dissociation and charge collection efficiencies and thus leading to high FFs. We also measured the relationship between the $J_{\rm sc}$ and the light intensity (*P*) to further understand the charge recombination property in the F–F-, F–Cl-, and F–Br-based devices. The relationship between them can be described with the formula of $J_{\rm ph} \propto P^{\alpha}$, where the exponential factor α is the recombination parameter. If there were no bimolecular recombination, the value of α is 1. The more the occurrence of bimolecular



Figure 3. AFM images for a) PBDB-T:F–H blend film, b) PBDB-T:F–F blend film, c) PBDB-T:F–Cl blend film, d) PBDB-T:F–Br blend film. TEM images for e) PBDB-T:F–H blend film, f) PBDB-T:F–F blend film, g) PBDB-T:F–Cl blend film, h) PBDB-T:F–Br blend film.







Figure 4. The GIWAXS patterns of F–H a), F–F b), F–Cl c), F–Br d) pure films. e) The in-plane line-cuts in GIWAXS for the pure films. f) The out-ofplane line-cuts in GIWAXS for the pure films.

recombination, the lower the values of the α ($\alpha \le 1$). As can be seen in the Figure 2d, the values of α are 0.957, 0.979, 0.989, and 0.996 with the F–H, F–F, F–Cl, and F–Br as the acceptors, respectively. All the values of α are very close to 1, and F–F, F–Cl-, and F–Br-based devices demonstrate higher α values, implying that less bimolecular recombination occurred in the

optimized devices of them and the results are consistent with their FFs.

Atomic force microscopy (AFM) in trapping mode and transmission electron microscopy (TEM) were used to further understand the morphologies of these blend films. From the AFM images in **Figure 3**a–d, the root-mean-square roughness values



of PBDB-T:F-F/F-Cl/F-Br blend films are 2.42, 3.48, and 3.45 nm, respectively, which are similar with PBDB-T:F-H (3.4 nm), indicating that all the three NFAs exhibit uniform and smooth surface morphologies. In the TEM images (Figure 3e-h), compared with the PBDB-T:F-H blend film, the blend films of F-F, F-Cl, and F-Br showed more distinctive fibrillar network. Especially, clear bicontinuous interpenetrating networks were observed in the F-Cl and F-Br films. These are favorable for exciton dissociation and charge transport, and thus result in high FFs. In order to further understand the improvement in performance with halogenation, GIWAXS was used. As shown in the Figure 4, F-H pure film shows weak (100) lamellar diffraction peak at ≈ 0.34 Å⁻¹ and $\pi - \pi$ stacking diffraction peak (010) at around 1.78 Å⁻¹, indicating that it is highly amorphous in the pure film. After introducing the halogen atoms to the end-group, F-F, F-Cl, and F-Br pure films exhibit strong (100) lamellar diffraction at around 0.34, 0.33, and 0.34 Å⁻¹, respectively, in the in-plane direction. The clear π - π stacking (010) diffraction peaks appeared at 1.82, 1.84, and 1.83 Å⁻¹ in the out-of-plane direction with the *d*-spacing of 3.45, 3.41, and 3.43 Å, respectively, implying the increased ordered packing and better crystallization of these three halogenated molecules. As can be seen in Figure S8 in the Supporting Information, compared with F-H blend film, the F-F, F-Cl, and F-Br blend films exhibit clear (010) diffraction peaks in the out-of-plane direction and clear (100) diffraction peaks in the in-plane direction, suggesting that these three blend films have preferred face-on orientation relative to the substrate. Such orientation is beneficial for efficient charge transport and collection. For F-Cl and F-Br blend films, the alkyl-alkyl regions show combined diffraction features from the donor and acceptor, which appeared as broad peaks and couldn't be easily separated. These features could be ascribed to the better mixing of donor and acceptor.

To further understand the superiority of F, Cl, and Br substitution, the charge mobilities of the optimized PBDB-T:F– F/F–Cl/F–Br were measured by the space-charge limited current method (Figure S4, Supporting Information). The hole and electron mobilities were measured with device structures of ITO/PEDOT:PSS/active layer/Au and ITO/ZnO/active layer/Al, respectively. As shown in Figure S4 in the Supporting Information, the hole and electron mobilities of PBDB-T:F–F/F–Cl/F–Br are $3.39 \times 10^{-4}/1.03 \times 10^{-4}$, $3.63 \times 10^{-4}/1.15 \times 10^{-4}$, $3.71 \times 10^{-4}/1.48 \times 10^{-4}$ cm⁻² V⁻¹ s⁻¹, respectively, which are in the same order but slightly improved from F to Br and significantly larger than those of PBDB-T:F–H ($3.37 \times 10^{-5}/2.40 \times 10^{-5}$ cm⁻² V⁻¹ s⁻¹).^[15] The enhanced and more balanced charge transport properties contributed to the higher J_{sc} and FF for the corresponding devices.

In summary, a series of NFAs, namely, F–F, F–Cl, and F–Br, with F, Cl, and Br substituted INCN as ending groups have been designed and synthesized. It was found that after the halogenation of the end-groups, the NFA molecules showed red-shifted and stronger absorptions, increased crystallinities, and higher charge mobilities. After blending with donor polymer PBDB-T, the F–F-, F–Cl-, and F–Br-based optimized devices exhibited PCEs of 10.85%, 11.47%, and 12.05%, respectively. Note that the device based on F–Br with PCE of 12.05% and remarkable FF of 76% is one of only a few OSCs with efficiencies over 12%

reported to date. Our results demonstrate that the halogenation is a promising strategy to delicately design and modify the NFAs for high-performance OSCs devices.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

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