### RESEARCH ARTICLE

# A thienyl-benzodithiophene-based two-dimensional conjugated covalent organic framework for fast photothermal conversion

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#### Abstract

Two-dimensional conjugated covalent organic frameworks (2D *c*-COFs) are emerging semiconductor materials for optoelectronic and photothermal applications. In particular, the highly tailorable, semiconducting thiophene-based 2D *c*-COFs have attracted considerable interest due to their nontrivial physicochemical properties such as photo-activity, broad optical absorption, tunable electronic structures, and so forth. Herein, we demonstrate a novel, crystalline 2D *c*-COF based on thienyl-functionalized benzodithiophene (BDT) and biphenyl (BP) via the Schiff-base polycondensation reaction. The resultant **BDT-BP-COF** exhibits a broad optical absorption up to ca. 600 nm and decent  $\pi$ -conjugation along the 2D polymer skeleton, as revealed by the optical absorption and theoretical calculations. The favorable  $\pi$ -conjugation and the abundant electron-rich thiophene units confer excellent photo-activity to **BDT-BP-COF** towards the usage of solar energy. As a proof-of-concept application, we explore **BDT-BP-COF** in photothermal conversion, in which it shows a fast surface-temperature increase upon light irradiation for seconds.

#### K E Y W O R D S

photothermal conversion, thienyl-benzodithiophene, two-dimensional conjugated covalent organic framework,  $\pi$ -conjugation

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#### 1 INTRODUCTION

Two-dimensional  $\pi$ -conjugated COFs (2D *c*-COFs) have emerged as a unique class of crystalline layer-stacked 2D conjugated polymers,<sup>1</sup> which are typically in-plane interconnected via  $\pi$ -conjugated linkages such as imines,<sup>2</sup> pyrazines,<sup>1b,3</sup> or vinylenes,<sup>1a,1c-1e,4</sup> while the out-of-plane interconnection is achieved by  $\pi$ - $\pi$  interactions between the aromatic moieties. Chemical tunability in the porous conjugated polymer backbone has enabled 2D c-COFs for broad electronic, optoelectronic and electrochemical applications such as organic field-effect-transistors,<sup>5</sup> organic lightemitting diodes,<sup>6</sup> organic solar cells,<sup>3b,7</sup> electrochromism,<sup>8</sup> photothermal conversion,<sup>9</sup> and so forth. Very recently, various electro-/photo-active building blocks such as pyrene,<sup>10</sup> porphyrin,<sup>11</sup> phthalocyanine,<sup>1b</sup> isoindigo,<sup>8,12</sup> thiophene and its derivatives (bithiophene, thienothiophene, benzodithiophene (BDT))<sup>4c,13</sup> have been incorporated into the 2D c-COF backbone. This has enabled nontrivial chemical and physical features such as electrical conductivity with high charge carrier mobility, photoactivity, and anisotropic charge transport.<sup>14</sup> In particular, the planar, polarizable, and tailorable thiophene units have conferred broad visible and near-infrared (NIR) optical absorption and highly tunable electronic structure to the resultant 2D c-COFs.<sup>15</sup>

Here, we report a novel, crystalline 2D c-COF based on thienyl-functionalized BDT and biphenyl (BP) linked by imine linkages (named as BDT-BP-COF). The assynthesized BDT-BP-COF exhibits an optical bandgap of 2.18 eV and effective  $\pi$ -conjugation along the 2D polymer skeleton, as confirmed by optical absorption spectroscopy and theoretical calculations. The good  $\pi$ -conjugation and the high density of electron-rich thiophene units confer excellent photoactivity to BDT-BP-COF. As a proof-of-concept application, we demonstrate the application of the new BDT-BP-COF in photothermal conversion, where it shows a fast surface-temperature increase upon light irradiation.

#### SYNTHESIS AND 2 **CHARACTERIZATION**

We first synthesized a novel aldehyde-substituted thienyl-BDT monomer (M1, with R = hexyl, Figure 1),

via a two-step organic reaction (see its synthetic details and characterizations in Scheme S1, Figures S1 and S2). The BDT-BP-COF was obtained via solvothermal Schiffbase 2D polycondensation between M1 and 4,4'diaminobiphenyl (M2) in an optimized solvent mixture of *n*-butanol and 1,2-dichlorobenzene (7:3 in v:v), using 6 M acetic acid as the catalyst (see other synthetic conditions in Table S1). The resulting COF polycrystals were collected and thoroughly washed with N,N-dimethylformamide, tetrahydrofuran, H<sub>2</sub>O, and acetone, affording the **BDT-BP-COF** as red powder up to 95% yield.

The successful Schiff-base 2D polycondensation is confirmed by Fourier transform infrared (FT-IR) spectroscopy, where we observed the disappearance of the amine signals  $(3100-3600 \text{ cm}^{-1})$  accompanied by the appearance of a new signal at  $1581 \text{ cm}^{-1}$ , which we assign to the C=N stretching vibration (Figure 2A). This is further confirmed by solid-state <sup>13</sup>C cross-polarization magic-angle spinning (CP-MAS) nuclear magnetic resonance (NMR) spectroscopy, where the peak at  $\sim$ 150 ppm can be attributed to the C atoms in the imine linkages (Figure 2B). Moreover, the predicted <sup>13</sup>C NMR spectrum in Figure S3 agrees well with the experimental result. X-ray photoelectron spectroscopy (XPS) displays the core levels of C 1s, N 1s and S 2p in BDT-BP-COF (Figure S4). The S 2p peak occurs at a binding energy in the range of 163-167 eV. Deconvolution of the signal generates S  $2p_{3/2}-2p_{1/2}$  doublet peaks at 165.2 and 164.0 eV, respectively, for the C-S from thiophene units (Figure S5).

Powder X-ray diffraction (PXRD) analysis confirms the crystalline nature of the BDT-BP-COF with distinct  $2\theta$  signals at 3.77°, 5.76°, 11.6°, 16.1°, and 26.1° (Figure 2C). The broad reflection at 26.1°, which is predominantly due to the  $\pi$ - $\pi$  stacking of **BDT-BP-COF** layers, indicates an interlayer distance of  $\sim$ 0.34 nm. The considerable twist in the biphenyl moieties could allow the two building blocks to connect, in principle, in two different topologies,<sup>16</sup> that is, a kagome or a rhombic framework (Figure 1). We thus modeled both possible COF structures in an eclipsed AA stacking mode using density functional theory (DFT) methods. The calculated PXRD patterns of the eclipsed AA-stacked kagome and rhombic BDT-BP-COF are shown in Figure 1B (see also



**FIGURE 1** Schematic synthesis of **BDT-BP-COF** in different topologies. R = hexyl. The structural models of rhombic and kagome **BDT-BP-COF** are shown on the right side. The lattice parameters are a = 21.3 Å, b = 24.6 Å, c = 3.9 Å,  $\alpha = 81.6^{\circ}$ ,  $\beta = 70.5^{\circ}$ ,  $\gamma = 118.8^{\circ}$  for the rhombic **BDT-BP-COF** and a = 46.3 Å, b = 46.3 Å, c = 6.0 Å,  $\alpha = 90^{\circ}$ ,  $\beta = 90^{\circ}$ ,  $\gamma = 60^{\circ}$  for the kagome **BDT-BP-COF**.

details in Figures S6 and S7). Although the kagome **BDT-BP-COF** produces a (120) reflection at  $2\theta = 3.81^{\circ}$  close to the experimental one at 3.77°, we could not conclude a preferred topology in the as-synthesized **BDT-BP-COF** based on mere PXRD.

Therefore, we performed N<sub>2</sub> physisorption measurement at 77 K aiming to explore the pore information of **BDT-BP-COF** (pore diameter of <2 or ~3 nm for rhombic or kagome topology, respectively, see details in Figure S8). The Type I isotherm indicates the existence of micropores (<2 nm) in **BDT-BP-COF** (Figure 2D, see the discussion of surface area with Figure S8). We obtained further the pore size distribution by fitting the experimental result with quenched solid DFT method. The result reveals a pore diameter of ca. 1.8 nm (shown in the inset of Figure 2D), which suggests a predominant rhombic topology in the as-synthesized **BDT-BP-COF**. However, the detection of a precise intra- and interlayer

arrangement in **BDT-BP-COF** represents a significant computational challenge stemming from the flexible polymer network and alkyl chains. To resolve the morphology of **BDT-BP-COF**, we measured scanning electron microscopy (SEM), which indicates aggregated flaky crystals (Figure S9). Moreover, thermalgravimetric analysis on **BDT-BP-COF** was performed in nitrogen, revealing excellent thermal stability without significant weight loss up to 450°C (Figure S10).

# 3 | OPTOELECTRONIC PROPERTIES AND PHOTOTHERMAL CONVERSION

To explore the optoelectronic property of **BDT-BP-COF**, we first calculated the electronic structures of the highest occupied molecular orbital (HOMO) and lowest



**FIGURE 3** Optoelectronic properties of **BDT-BP-COF**. (A) The electronic structure of HOMO and LUMO of the representative structural unit of **BDT-BP-COF**. (B) UV-visible absorption of **BDT-BP-COF** in 2-propanol and corresponding Tauc plot profile shown in the inset. (C) Fluorescence ( $\lambda_{ex} = 400$  nm) spectrum of **BDT-BP-COF**.

unoccupied molecular orbital (LUMO) of a representative structural unit of **BDT-BP-COF** (named as **M3**, see the chemical structure in Figure 1). **M3** is a wide-bandgap semiconductor with HOMO and LUMO energy levels at -5.21 and -2.46 eV, respectively. Clearly, **M3** presents delocalized HOMO electrons throughout the thienyl-BDT, imine and benzene units, which corresponds to a decent  $\pi$ -conjugation in the 2D plane in **BDT-BP-COF** (**Figure 3**A). It is noteworthy that an asymmetric LUMO

appears with an obvious distribution along the linear BDT rather than the thienyl-benzene direction, implying superior  $\pi$ -conjugation in the BDT-bridged polymer strand. The UV-visible absorption of **BDT-BP-COF** was then measured by dispersing the powder sample in 2-propanol. The recorded spectrum displays a broad and intense absorption in the range of ca. 400–600 nm with an absorption edge located at 581 nm (Figure 3B). The optical bandgap extracted from the Tauc plot is ~2.18 eV

**FIGURE 2** Chemical and crystal structures of **BDT-BP-COF**. (A) FT-IR spectra of B**DT-BP-COF** and the starting monomers. (B) <sup>13</sup>C CP-MAS NMR spectrum. (C) Experimental and calculated PXRD patterns. (D) N<sub>2</sub> adsorption-desorption isotherm and pore size distribution.



**FIGURE 4** Photothermal conversion property of **BDT-BP-COF**. (A) Photothermal images of **BDT-BP-COF** pellets before and after 1 sun illuminations. (B) Cycling experiment.

(see also the UV-visible absorption spectrum measured in the solid state in Figure S11). Moreover, the fluorescence spectrum of the **BDT-BP-COF** dispersion indicates emission maxima at 607 nm.

Taking the broad optical absorption, the pronounced  $\pi$ -conjugation and the rich photo-active thiophene units into consideration, BDT-BP-COF possesses great potential in the utilization of solar energy, that is, photorelated applications. As a proof-of-concept, we evaluated the photothermal conversion behavior of BDT-BP-COF. materials and conjugated polymers In carbon (e.g., polyaniline, polypyrrole<sup>17</sup>), the energy absorbed by the electrons relaxes by means of electron-phonon couplings. This avoids the re-emission of photons at low temperatures; on the contrary, the energy of incident photons will transfer to vibrational modes within the atomic lattices, leading to a temperature increase in the material.<sup>18</sup> To prepare the photothermal device from BDT-BP-COF, the COF powder sample was pressed into a round pellet, and further placed onto a quartz glass substrate (see details in Experimental Section and Figure S12). Upon standard solar radiation (i.e., AM 1.5G solar spectrum, 100 mW cm<sup>-2</sup>),<sup>19</sup> we monitored the surface temperature variation of the COF pellet by IR camera. It is apparent that the surface of BDT-BP-COF shows a sharp increase in temperature from 28.3 to  $\sim$ 32°C after light irradiation for only a few seconds (Figure 4A,B). Extending to 3 min, the temperature reached 41.5°C with a difference of 13.2°C compared to

the ambient one, indicating the excellent solar-thermal conversion efficiency of **BDT-BP-COF**, while the blank quartz glass substrate exhibited merely an increase of 4.2°C under the same conditions. We further tested the photothermal stability of **BDT-BP-COF** by a three-cycle experiment (Figure 4B). Though the background slightly rose we observed almost no change in the time-resolved temperature curve after long-term irradiation.

# 4 | CONCLUSION

In conclusion, we have demonstrated a novel semiconducting imine-linked 2D *c*-COF based on the large  $\pi$ -conjugated thienyl-BDT building block. The achieved **BDT-BP-COF** exhibits broad optical absorption and decent 2D  $\pi$ -conjugation, and thus excellent photothermal conversion ability. Further efforts to synthesize related vinylene-linked 2D *c*-COFs, that is, 2D poly(arylene vinylene)s with boosted 2D  $\pi$ -conjugation will be explored to enhance the charge carrier-transport ability and extend the optical absorption further into the NIR region thus rendering advanced conjugated semiconducting materials for high-performance photothermal or optoelectronic devices.

# 5 | EXPERIMENTAL SECTION

# 5.1 | Synthesis of BDT-BP-COF

Monomer **M1** was synthesized according to a literature procedure.<sup>20</sup> A 5 mL high-pressure glass tube was charged with **M1** (12.00 mg, 0.019 mmol), **M2** (9.48 mg, 0.038 mmol), dichlorobenzene/*n*-butanol/6 M acetic acid (0.3/0.7/0.1 mL). The tube was sonicated at room temperature for 5 min, degassed by three freeze-pump-thaw cycles, sealed under vacuum and heated at 120°C for 2 days. After cooling to room temperature, the precipitate was filtrated, washed with dimethylformamide, H<sub>2</sub>O, methanol and acetone, respectively, collected, and dried under vacuum at 100°C overnight to give red powders in 95% yield.

# 5.2 | Photothermal conversion

The **BDT-BP-COF** powders were pressed into a round pellet with a thickness of 0.35 mm and diameter of 0.6 cm. The pellet sample was placed on a quartz glass substrate  $(3 \times 3 \times 3 \text{ mm}^3)$  for the photothermal study. The fabricated device was irradiated with sunlight under a solar simulator (AM 1.5G solar spectrum, 100 mW cm<sup>-2</sup>), while the temperature was monitored by an infrared camera.

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