

# Direct Photopatterning of BODIPY-Based Small Molecules via Thiol-ene Click Chemistry

Mücahit Özdemir,<sup>[a]</sup> Sinem Altınışik,<sup>[b]</sup> İpek Ömeroğlu,<sup>[c]</sup> Baybars Köksoy,<sup>[d]</sup> Mahmut Durmuş,<sup>[c]</sup> Bahattin Yalçın,<sup>\*[a]</sup> and Sermet Koyuncu<sup>\*[b]</sup>

**Abstract:** We demonstrate a BODIPY-based small molecule photopatterned surface by thiol-ene click chemistry at room temperature utilizing only 366 nm UV light. The resulting cross-linked polymer exhibits porous surfaces according to AFM and TEM results. A dramatic blue shift occurred in absorption spectra and the photoluminescence was also

intensified as a result of crosslinking. Optical and electrochemical results are compatible with the DFT calculations. The obtained results prove that the photopatterned BODIPY-based material can be easily and inexpensively applied in multilayer optoelectronic devices.

## Introduction

Crosslinkable polymers have been very popular since the 1970s thanks to their many outstanding properties such as high stability and multi-layer film processing and one of the most important reactions used to produce crosslinked polymers is the thiol-ene click reaction.<sup>[1]</sup> The most optimal homogeneous network topologies generated from these systems are cross-linked polymers with extremely low polymerization shrinkage stress. The overall simplicity, stability, and mechanism of the photoactive thiol-ene click reaction arrangement is a controllable homogeneous polymer network with significantly simplified reaction kinetics and resistance to oxygen inhibition.<sup>[2]</sup> The reaction procedure eliminates some of the disadvantages like requirements of high temperature and metal catalysis associated with traditional light-triggered polymer reaction systems. Recently, these distinct advantages have made the focus on extensive new research and practical applications of the photoactive thiol-ene click reaction.<sup>[3]</sup> Further, photopatterning is a novel and popular modeling technique that involves selectively exposing a photosensitive polymer to light through a mask, leaving a latent image in the polymer that may be selectively dissolved to enable patterned access to an underlying

substrate.<sup>[4]</sup> Photopatterning *via* thiol-ene *click* reaction can be used to form polymer thin films.<sup>[5]</sup> The crosslinking reaction is carried out simply and easily under mild conditions and under UV light. It has been revealed that the cross-linked films which are obtained with the help of a thiol-ene click have several advantages compared to other cross-linked polymer films, such as the absence of high-temperature requirements and the absence of unwanted by-products.<sup>[6]</sup>

It has been observed that there were/have been many studies on the stability of cross-linked thin films produced with electroactive polymers such as thiophene and fluorene, especially in optoelectronic applications such as OLED, OFET, electrochromic, photovoltaic.<sup>[7]</sup> Carter *et al.* used thiol-ene *click* chemistry for the first time to produce crosslink polyfluorene thin films using 4-phenylthienyl end groups.<sup>[8]</sup> It has also been shown that these cross-linked films are completely suitable for the fabrication of polymeric light-emitting diodes. At the same time, the photo masking (photopatterning) process was also applied to PLED devices obtained from cross-linked polymer films, and thiol-ene chemistry could also be used for photo masking. The electrical and optical properties of two-component combinations of electron and hole transfer materials were used in single and double-layer organic light-emitting diodes were investigated by Davis *et al.* (2011).<sup>[8]</sup> Photo effect patterning on the surface was carried out by the Reynolds and Krebs research groups employing a 1 mm<sup>2</sup> mask on a polymer containing acrylate side groups. It is stressed that the technique utilized in the study is particularly effective for signboards.<sup>[9]</sup>

BODIPY (4,4-difluoro-4-bora-3a,4a-diaza-s-indacene) as a synthetic dye exhibits effective fluorescence, and provides high fluorescence quantum efficiency values, it has high molar absorption coefficients and exhibits unique photophysical properties such as thermal and photostability.<sup>[10]</sup> These properties of BODIPY dyes allow them to show high-intensity light emission and be used as electron transfer agents in various applications.<sup>[11]</sup> BODIPY dyes have the potential to be a scaffold or building block in complex polymer structures in addition to being a high-specification chromophore/fluorophore with their unique arrangement of substituent groups kept at fixed

[a] M. Özdemir, Prof. Dr. B. Yalçın  
Department of Chemistry  
Marmara University  
34722 Istanbul (Turkey)  
E-mail: byalcin@marmara.edu.tr

[b] S. Altınışik, Prof. Dr. S. Koyuncu  
Department of Chemical Engineering  
Çanakkale Onsekiz Mart University  
17100 Çanakkale (Turkey)  
E-mail: skoyuncu@comu.edu.tr

[c] Dr. İ. Ömeroğlu, Prof. Dr. M. Durmuş  
Department of Chemistry  
Gebze Technical University  
41400 Kocaeli (Turkey)

[d] Assoc. Prof. Dr. B. Köksoy  
Department of Chemistry  
Bursa Technical University  
16310 Bursa (Turkey)

angles.<sup>[12]</sup> In addition, BODIPY-containing and conjugated small molecules and polymers containing BODIPY skeletal structure in repeated units in the literature are important for electronic device applications and especially for electrochromic and photovoltaic materials.<sup>[13]</sup> BODIPY compounds absorb and emit light at wavelengths less than 600 nm when they are not modified. Extra donor materials such as thiophene allows BODIPY derivatives to be used as absorbers in near-infrared applications due to increase the conjugation.<sup>[14]</sup>

There are limited studies on BODIPY's photopatterned films and they were obtained with laser support.<sup>[15]</sup> In this study, photopatterned fluorescent surfaces *via* thiol-ene click chemistry have been obtained by using electroactive BODIPY as a small molecule in simple and mild conditions for the first time. According to the AFM measurements, it was observed that more porous films were obtained after crosslinking process. In addition, the effect of cross-linking on optical and electrochemical properties has been investigated. The obtained cross-link structure was determined by the 3D molecular geometry and HOMO-LUMO charge distribution using DFT calculations. It was observed to be quite compatible when compared to the experimental data. The results show that BODIPY-based cross-linking films are particularly suitable for multilayer and patterned optoelectronic device applications.

## Results and Discussion

The DSB was synthesized in three steps (Figure 1) and structurally characterized by common spectroscopic methods such as FT-IR, MALDI-TOF, and NMR (<sup>1</sup>H-, <sup>13</sup>C-, <sup>11</sup>B-, <sup>19</sup>F-). All spectra were given in the supporting information file and supported the proposed structure of DSB.

FT-IR technique was used to prove the obtaining cross-linked polymer (PDSB) on the film surface. The carbonyl peak at the tetrathiol-based molecule used as a linker among styrene units of DSB was clearly observed at 1729 cm<sup>-1</sup>. In addition, the C–S bond formed due to the opening of the styrene moiety at DSB was observed at 1140 cm<sup>-1</sup> (Figure 2a). The presence of these two peaks observed when compared to the FT-IR spectrum of the DSB monomer is clear evidence of crosslinking polymer obtained.

TGA analysis was measured under a nitrogen atmosphere in the range of 25–800 degrees with 10 degrees increments per minute. In the TGA thermogram, there were 2 dramatic masses; the first one was a 10% mass loss from 197 °C to 291 °C, and it occurred by the rupture of alkene groups. The second one was a 30% mass loss from 385 °C to 530 °C, and it occurred when the phenyl groups which alkene moiety removed previously, attached to the 2nd and 6th carbons were broken. As a result, TGA analysis showed that the presence of nearly 50% residues at 800 degrees is an indication that the molecule is quite stable at high temperatures (Figure 2b). The 5% weight loss temperature of the crosslinked polymer (PDSB) was found to be above

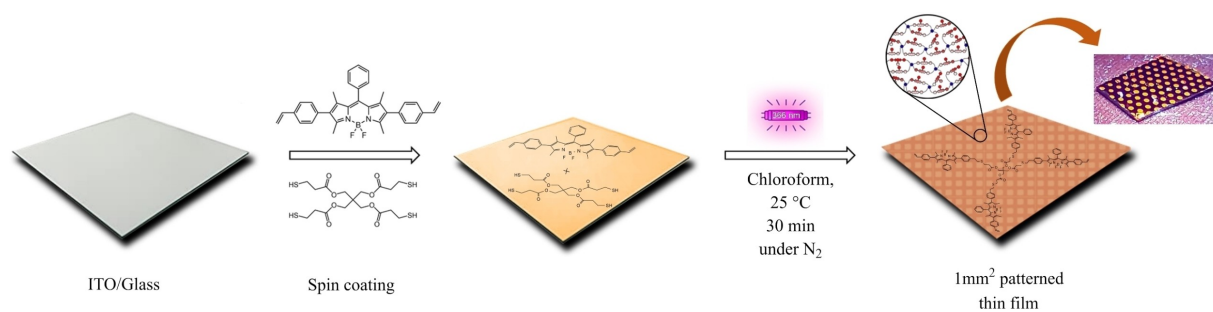


Figure 1. Preparation of cross-linked films containing a BODIPY derivative.

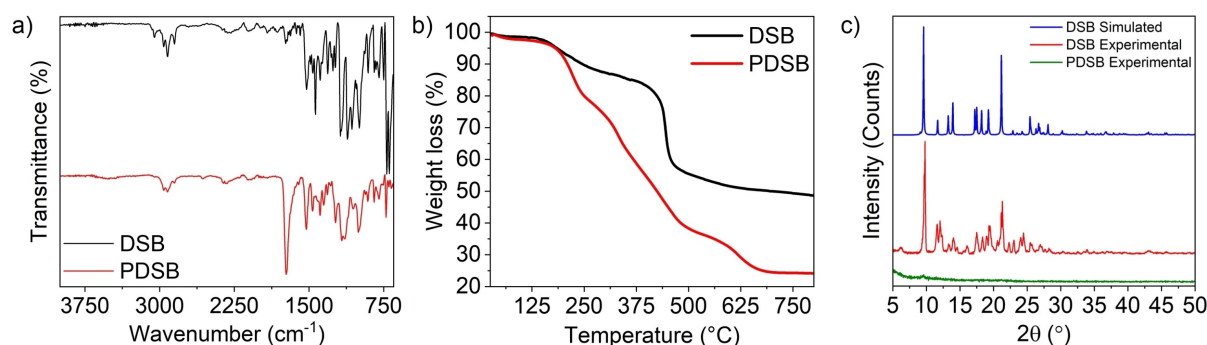


Figure 2. A) Comparative FT-IR spectra of DSB and PDSB compounds. B) The TGA curves of DSB and PDSB. C) Experimental and simulated XRD patterns of monomer (DSB) and crosslinked BODIPY (PDSB).

200 °C and this weight loss was attributed to the unreacted monomers and residual solvent remaining in the polymer matrix. In the cross-linked polymer, the initial degradation temperature is in the range of 200–275 °C and above this temperature, it exhibited two main degradation steps. Due to the structure of the alkyl chains, more mass losses are observed compared to the monomer due to the breakage of the flexible bridges. Although the flexible alkyl chains increased the thermal decomposition temperature, the reduced crosslinking led to an increase in the yields of charcoal obtained.

As a result of the powder XRD analysis, it was seen that the monomer BODIPY (DSB) has a crystalline structure, and the polymeric structure formed on the surface as a result of crosslinking turns into an amorphous structure due to the flexible feature of the crosslinker (Figure 2c). SEM images support the transformation from crystalline to amorphous behavior as a result of crosslinking (Figure 7).

To understand the geometrical/structural properties of 2,6-distyrene-substituted BODIPY precursor and its crosslinking polymer, they were optimized using the density functional theory method (DFT), and HOMO-LUMO energy levels/orbital contributions were calculated using time-dependent density functional theory (TD-DFT) with B3LYP basis set (Figure 3).

According to these results, the charges separated on the whole conjugated BODIPY-styrene donor skeleton at the HOMO were concentrated to BODIPY center acceptor moieties at

LUMO due to the high electronegativity of the fluorine center of the BODIPY core. In addition, it was observed that charges were localized in terminal styrene groups at the HOMO-1 and LUMO + 1 orbitals. In the crosslinked BODIPY compound, on the other hand, the electrons are not on the crosslinker, but on the BODIPY core, similar to its behavior in the monomer, and the calculated band gap has not changed much, as in the experimental results.

In the DFT calculation using the crystal data, the styrene groups attached to the molecule from the 2 and 6 positions are cross positioned to each other (torsion angle 57.06° with the pyrrole ring), and the phenyl ring in the meso position is orthogonal to these rings (83.42°), similar to the BODIPY compounds in the literature. Considering the TD calculations of the target BODIPY compound in three different solvents, the main transition ( $S_0 \rightarrow S_1$ ) gives approximately the same bandgap (2.90 in DMSO, 2.89 in THF, and 2.88 eV in Toluene). As for the orbital contributions, the contribution to the main transition in DMSO ( $\lambda_{\max} = 468$  nm, oscillator strength = 0.7783) and THF ( $\lambda_{\max} = 469$  nm, oscillator strength = 0.7847) solvents are 92% to HOMO  $\rightarrow$  LUMO and 8% to HOMO-2  $\rightarrow$  LUMO. In toluene ( $\lambda_{\max} = 471$  nm, oscillator strength = 0.8276) it was determined as 93% for HOMO  $\rightarrow$  LUMO, and 7% for HOMO-1  $\rightarrow$  LUMO.

The absorption and emission of thin films prepared by the spin coater method were investigated by UV-Vis spectrophotometer as well as spectrofluorometer. The absorption of the

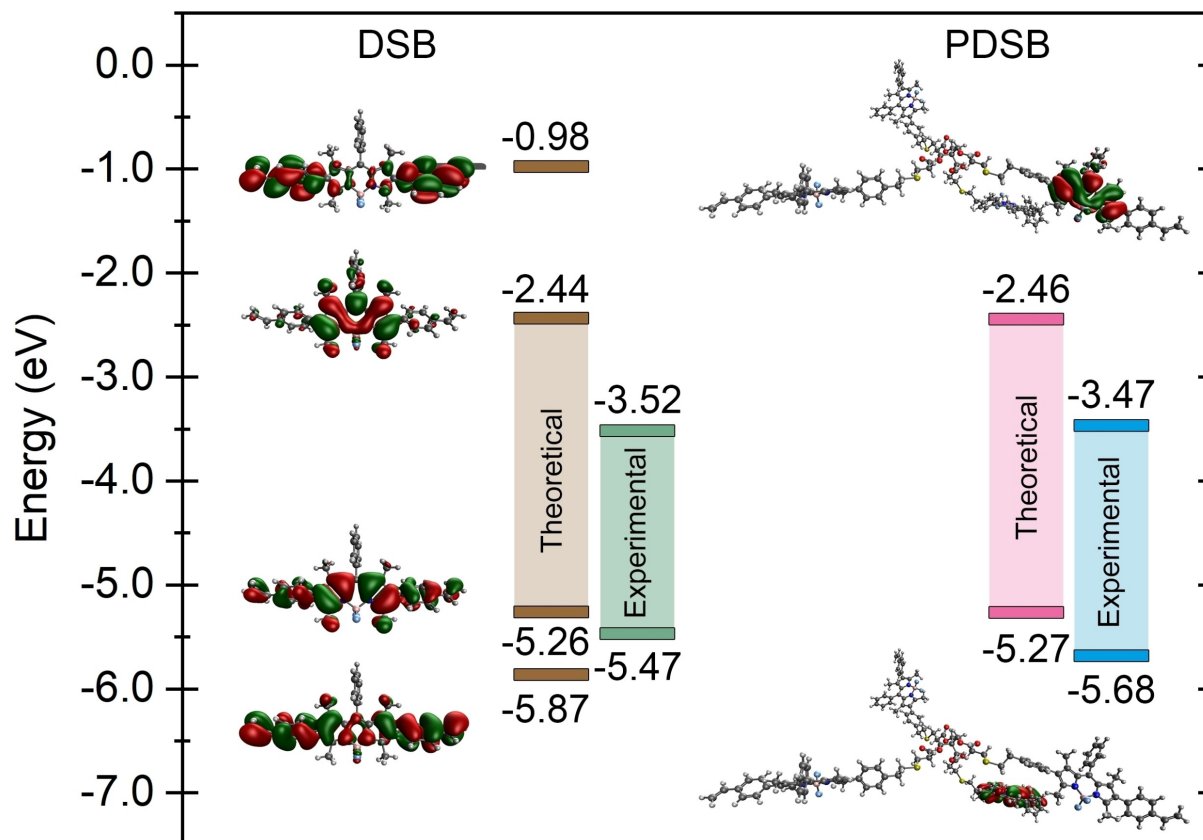


Figure 3. Molecular orbital diagram of styrene-BODIPY.

bare film of **DSB** (without crosslinker) was observed centered at about 575 nm. As a result of the cross-linking, it was observed that the absorption band of the film at around 540 nm showed a 35 nm blueshift because of the decreased  $\pi$ -conjugation after the cross-linking process. In addition, it was observed that the band intensity at 540 nm decreased when the crosslinker ratio was increased.<sup>[8]</sup> When excited from the absorption maxima, it was observed that the **DSB** molecule has an emission in the near-infrared region centered at about 750 nm. After cross-linking process to obtain **PDSB**, the remarkable blueshift (170 nm) and more intense emission were observed.<sup>[16]</sup> This can be due to the terminal alkene moiety being broken as a result of cross-linking and thus the conjugation is narrowed. At the same time, the fluorescence emission was intensified by the donor sulfur atom at the tetrathiol-based crosslinker (Figure 4).

CV and DPV techniques were used for the electrochemical characterization of the **DSB** and **PDSB** (Figure 5). In the CV of **DSB**, reversible oxidation at around 1.25 V was observed due to the conjugated BODIPY styrene moiety in the core structure. Besides, reversible reduction of the difluoro BODIPY center was observed at around  $-1.10$  V. In addition, the fully charge-discharge behavior of **DSB** in both anodic and cathodic regions was proved by DPV measurement. On the other hand, after the crosslinking process, both oxidation and reduction waves shifted to high potential due to the tetrathiol-based crosslinker containing electron-accepting carbonyl moieties. According to these results, the HOMO-LUMO bandgap calculated from the oxidation and reduction onset potential was found to be  $-5.47$  and  $-3.52$  eV ( $E_g = 1.95$  eV), respectively. While the LUMO position did not change much after crosslinking (LUMO =  $-3.47$  eV), the HOMO position decreased considerably (HOMO =  $-5.68$  eV) as the oxidation potential shifted to a higher potential. Accordingly, the electrochemical bandgap expanded from 1.95 to 2.21 eV. Based on the outcome that the **DSB** compound exhibits an ideal ambipolar behavior, proving that it can be used for optoelectronic applications.

When the AFM image of the bare **DSB** thin film prepared from its solution without a crosslinker is examined, it is seen

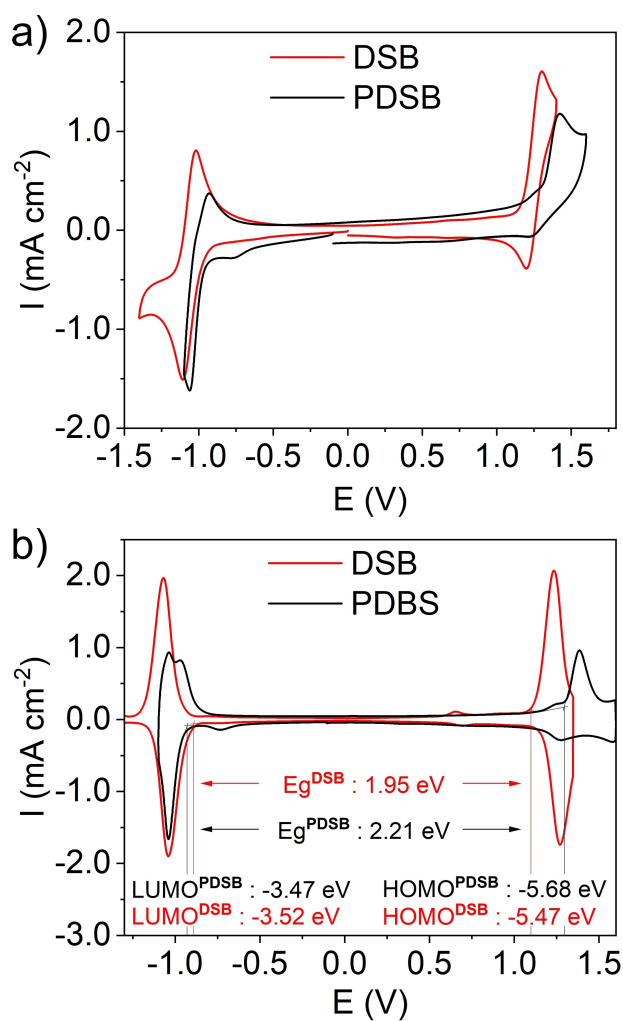


Figure 5. The cyclic voltammetry (a) and differential pulse voltammetry (b) voltammograms of **DSB** and **PDSB** in 0.1 M TBAPF<sub>6</sub>/DCM electrolyte solution at a scan rate of 100 mV/s Ag wire.

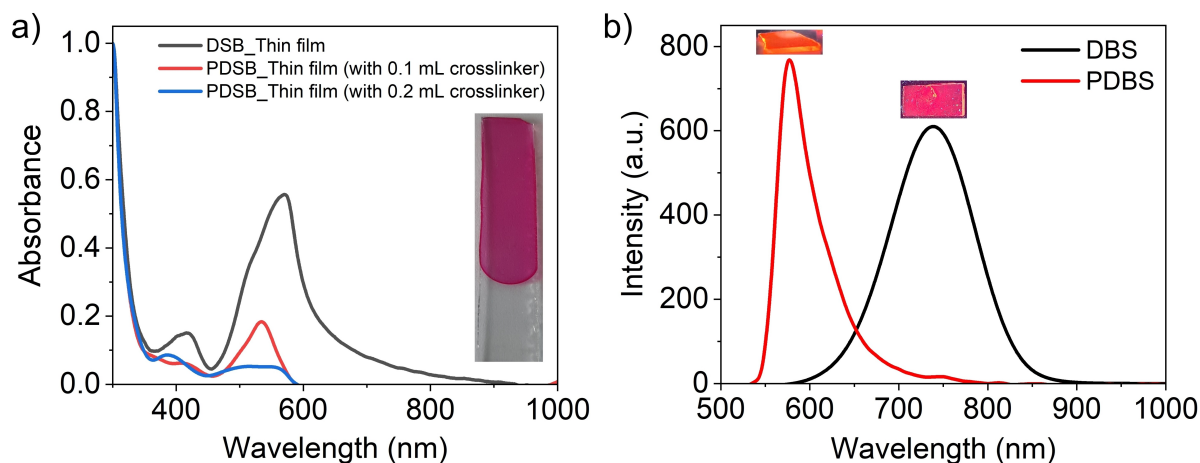


Figure 4. UV-Vis absorption (a) and fluorescence emission (b) spectra of **PDSB** on thin film.

that it has a very smooth surface (Figure 6). The smooth surface of bare DSB film is due to the planar structure of the BODIPY core. However, some defects were observed on the film surface because of the active vinyl group on the styrene subunit. In addition, the samples were prepared for TEM measurements by drop-casting method from the chloroform solution onto the grid. Some spots seen in the AFM images were also clearly observed in the TEM. When the AFM image of the films prepared in the presence of a crosslinker is examined, it was seen that agglomerations occur on the film surface and a rougher surface is formed.

When the TEM images are examined, particles between 0.5 and 2  $\mu\text{m}$  diameter are observed due to aggregation caused by crosslinking. According to these results, it was observed that

the RMS roughness increased from 3.44 nm to 6.47 nm after the crosslinking process.

On the other hand, Scanning Electron Microscopy (SEM) plots show that the monomer has a wide range of crystal domains. As a result of cross-linking, it was observed that these crystal domains became smaller with the introduction of the flexible tetrathiol-based crosslinker into the structure. In addition, Energy Dispersive X-Ray analysis (EDX) was also performed, and it was determined that oxygen and sulfur atoms in the structure of the crosslinker were observed in the spectrum (Figures S12–13).

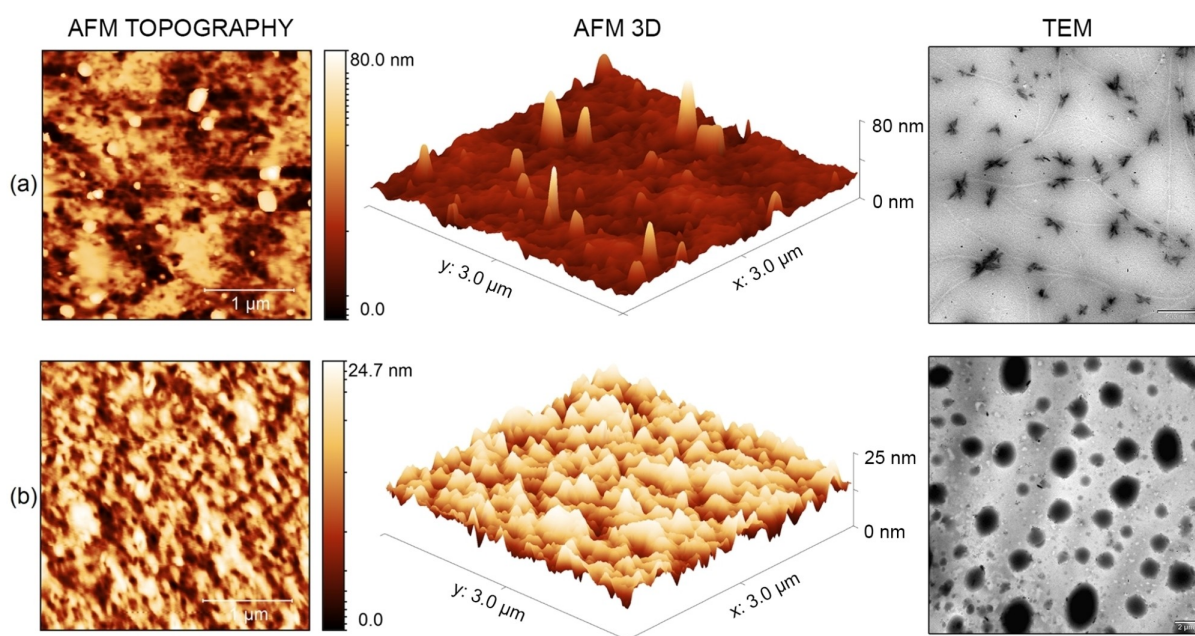


Figure 6. AFM and TEM images of DSB (a) and PDSB (b) films.

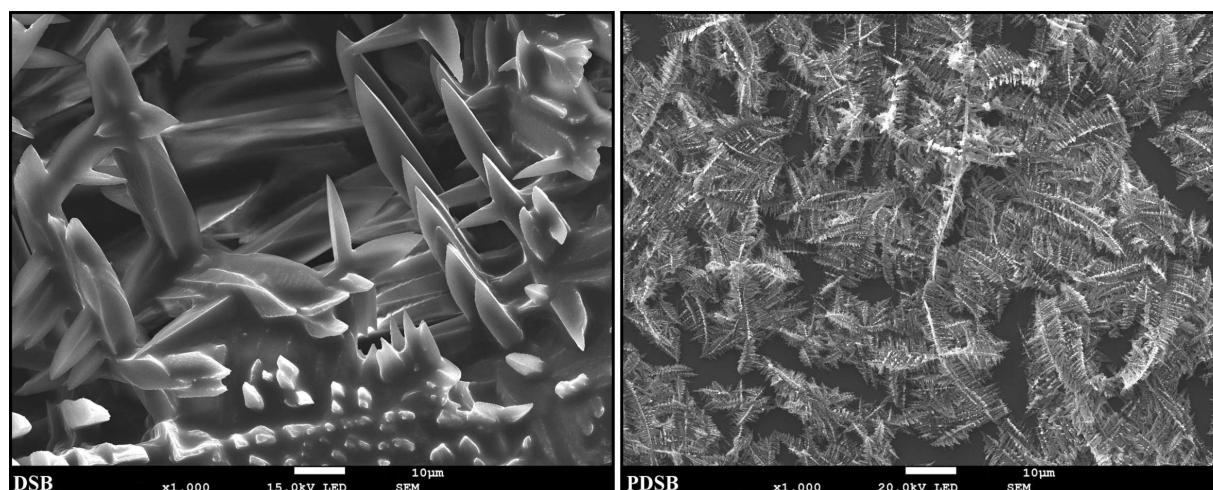
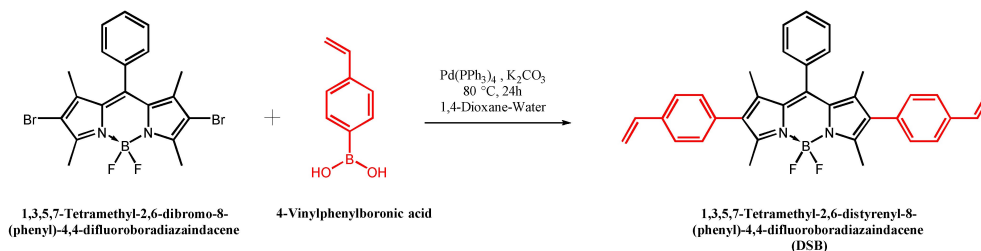
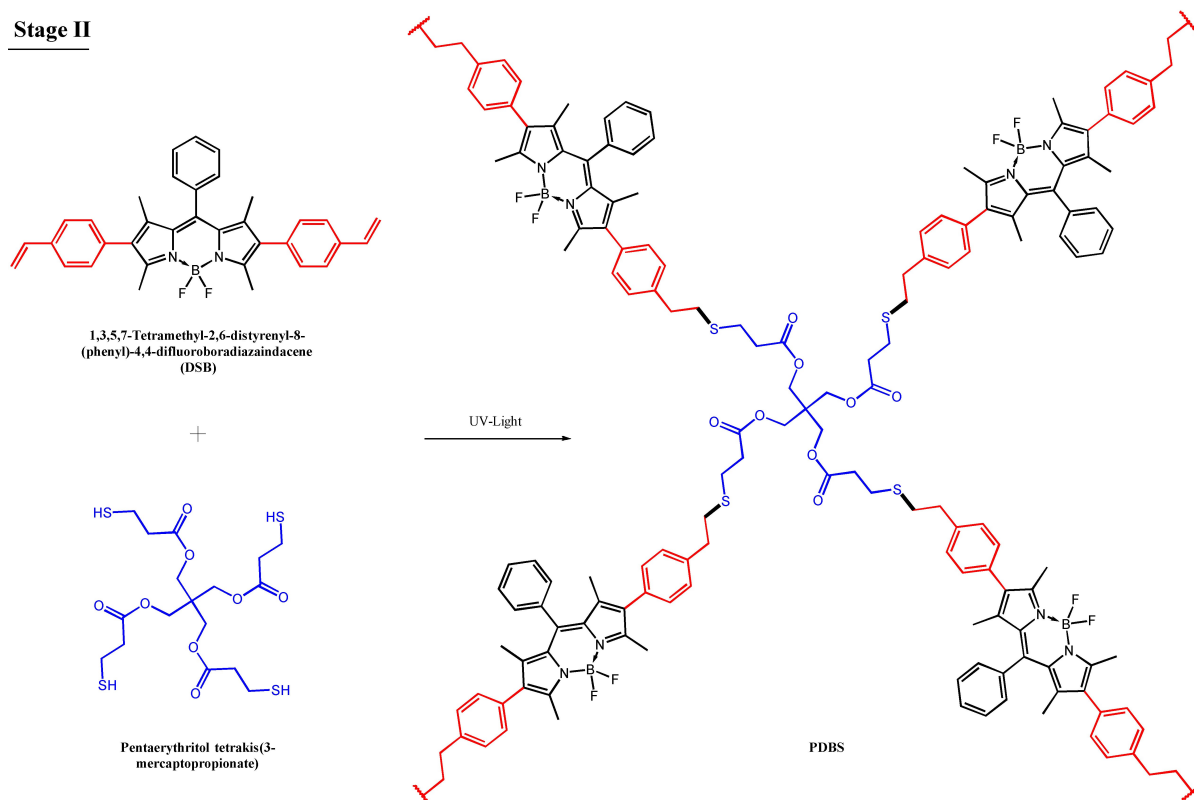


Figure 7. SEM images of DSB (a) and PDSB (b) films.

### Stage I



### Stage II



**Scheme 1.** The synthetic pathway of 1,3,5,7-Tetramethyl-2,6-distyrenyl-8-(phenyl)-4,4-difluoroboradiazaindacene (DSB) and its cross-linked polymer (PDSB).

## Conclusion

In summary, the distyrene-bearing BODIPY chromophore was synthesized and direct photopatterning was performed on the film surface by thiol-ene click chemistry. After crosslinking process, the  $\pi$ -conjugation decreased with the breaking of the terminal styrene groups on BODIPY, and accordingly, it was observed that the band gap ( $E_g$ ) increased as a result of both optical and electrochemical measurements. It was observed that the theoretical DFT calculations were compatible with the experimental results, and as a result of the 3D modeling study, it was seen that the lattice structure was formed by cross-linking. Besides, according to AFM and TEM measurements, both the roughness increased, and agglomeration was observed on the surface. SEM images and XRD results support the decrease of the crystallinity as a result of crosslinking process on the thin film surface. Finally, this method for BODIPY-based

small molecules is suitable for fabricating multilayer and patterned optoelectronic devices by an entire solution process.

## Experimental Section

### Synthesis

1,3,5,7-Tetramethyl-8-(phenyl)-4,4-difluoroboradiazaindacene and 1,3,5,7-tetramethyl-2,6-dibromo-8-(phenyl)-4,4-difluoroboradiazaindacene were synthesized according to the literature.<sup>[17]</sup> The synthetic pathways for 1,3,5,7-tetramethyl-2,6-distyrenyl-8-(phenyl)-4,4-difluoroboradiazaindacene (DSB) and its cross-linked polymer (PDSB) were showed in Scheme 1.

### 1,3,5,7-Tetramethyl-2,6-distyrenyl-8-(phenyl)- 4,4-difluoroboradiazaindacene (DSB)

90 mg (0.186 mmol) 2,6-dibromo-8-(phenyl)-4,4-difluoroboradiazaindacene, 82.89 mg (0.560 mmol) 4-vinylphenylboronic acid and 129 mg (0.93 mmol)  $K_2CO_3$  were dissolved in 5 mL dioxane:water (4:1) solution. The reaction mixture was stirred in an argon atmosphere for 15 minutes. Then, 22 mg (0.019 mmol)  $Pd(PPh_3)_4$  was added to this reaction mixture, and it was stirred at 80 °C under an argon atmosphere for 24 hours. At the end of this period, the reaction mixture was cooled to room temperature. This mixture was extracted several times with dichloromethane (DCM) and the solvent was evaporated. The crude product was purified by column chromatography using silica gel as an absorbent and DCM-hexane (1:1) as an eluent. Chemical formula  $C_{35}H_{31}BF_2N_2$ , Yield: 80.6 mg (82.0%); Anal. calcd. for  $C_{35}H_{31}BF_2N_2$ : C 79.55, H 5.91, N 5.30% – found: C 79.52, H 5.28, N 5.27%; FT-IR ( $n_{max}/cm^{-1}$ ): 3043 (Ar–CH), 2986–2846 (Alp–CH), 1545–1512 (C=C); UV-vis (THF,  $\lambda_{max}/nm$ )(log $\epsilon$ ): 381(3.08), 533(3.89);  $^1H$ -NMR ( $d_6$ -DMSO, 500 MHz,  $\delta$  ppm): 7.51–7.46 (m, 3H, meso-phenyl-H), 7.43 (d, 4H,  $J=8.18$  Hz, styrene-H), 7.34 (dd, 2H,  $J=7.99$  and 1.82 Hz, meso-phenyl-H), 7.12 (d, 4H,  $J=7.97$  Hz, styrene-H), 6.72 (dd, 2H,  $J=17.6$  and 10.9 Hz, alkene-H), 5.75 (d, 2H,  $J=17.6$  Hz, alkene-H), 5.26 (d, 2H,  $J=10.9$  Hz, alkene-H), 2.54 (s, 6H,  $-CH_3$ ), 1.30 (s, 6H,  $-CH_3$ );  $^{13}C$ -NMR ( $d_6$ -DMSO, 125 MHz,  $\delta$  ppm): 154.3, 142.1, 139.2, 136.5, 136.3, 135.4, 133.4, 133.2, 131.4, 130.4, 129.3, 129.1, 128.1, 126.2, 114.0, 13.4, 12.7;  $^{11}B$ -NMR ( $d_6$ -DMSO, 160 MHz,  $\delta$ , ppm): 0.99 (t,  $J=32.7$  Hz),  $^{19}F$ -NMR ( $d_6$ -DMSO, 470 MHz,  $\delta$ , ppm):  $-146.1$  (q,  $J=32.6$  Hz); MALDI-TOF (m/z): Calc. 528.45 for  $C_{35}H_{31}BF_2N_2$ , found 528.59 as  $[M]^+$ .

### Preparation of cross-linked films (PDSB)

Pentaerythritol tetrakis(3-mercaptopropionate) crosslinker was added to the solution of styrene-BODIPY compound (DSB) in chloroform at different ratios and thin films were prepared. As a result of the reaction carried out with the application of 366 nm 100 W UV light for 15 minutes on the film surface, the films were washed with methanol. It was determined that the crosslinked surface remained on the surface without dissolving.

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### Conflict of Interest

The authors declare no conflict of interest.

### Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

**Keywords:** BODIPY · photopatterning · thiol-ene click · electrochemistry · surface characterization

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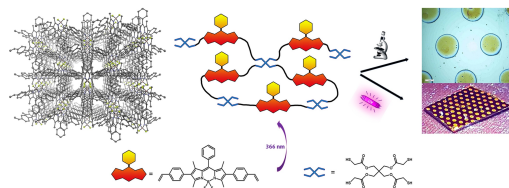
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## RESEARCH ARTICLE



**A BODIPY-based small-molecule photopatterned surface** by thiol-ene click chemistry was prepared at room

temperature using only 366 nm UV light.

*M. Özdemir, S. Altınışik, Dr. İ. Ömeroğlu, Assoc. Prof. Dr. B. Köksoy, Prof. Dr. M. Durmuş, Prof. Dr. B. Yalçın\*, Prof. Dr. S. Koyuncu\**

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**Direct Photopatterning of BODIPY-Based Small Molecules via Thiol-ene Click Chemistry**