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Synthesis and Characterization of 3,6 - Disubstituted Carbazole Containing Fluorene and DSSC Applications

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Abstract: Heterocyclic compounds containing nitrogen are regarded as essential structural motifs present in numerous natural products and organic materials. Within this group, carbazoles are important hetero-aromatic molecules which play a significant role in the development of materials with various functionalities. Carbazole derivatives play a crucial role in various organic electronic devices such as organic transistors (OTFTs), organic light-emitting diodes (OLEDs), and organic solar cells. In this study, the solubility of the carbazole nucleus, which initially had limited solubility in organic solvents, was increased by adding an octyl group. Next, a carbazole-based compound 3,6-di(fluorene-9)-9-octyl-9H-carbazole (IV) was synthesized via the Suzuki-Miyaura cross-coupling reaction (Figure 1). The photophysical and thermal properties of this compound were determined by UV-Vis, thermogravimetric analysis (TGA) and differential thermal analysis (DTA). The HOMO and LUMO energy levels and the optical band gap (E_g,opt) were obtained by cyclic voltammetry (CV) and absorption bands. TiO_2-based dye-sensitized solar cells (DSSCs) were fabricated using compound IV. The photoelectrochemical properties of the resulting TiO_2-DSSCs were measured.

Keywords: Carbazole, Suzuki-Miyaura cross-coupling reactions, Cyclic Voltammetry, DSSC.

Introduction

Carbazole is a class of aromatic ring compounds with the molecular formula $C_{12}H_9N$, containing nitrogen, and is associated with conjugated structures known for their electron and charge transfer properties (Bouaziz et al., 2015; Behrens, 1900). Carbazole was first discovered by Graebe and Glaser in 1872 when they isolated it from coal tar (Bouaziz et al., 2015). Carbazole exhibits strong fluorescence when exposed to ultraviolet (UV) light, a characteristic that makes it frequently used in the production of photographic plates and in paint manufacturing (Drechsel, 1858). This compounds can be substituted at the nitrogen position, and they can be substituted at the 3- and 6-positions to yield 3,6-carbazoles or at the 2- and 7-positions to yield 2,7-carbazoles (Doskocz et al., 2006). Fluorene is a white crystalline hydrocarbon compound known for its strong fluorescence properties. Additionally, fluorene is found in small quantities in coal tar and can also be synthetically produced, with a chemical formula of $C_{13}H_{10}$. Fluorene belongs to a group of chemicals known as polycyclic aromatic

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hydrocarbons (PAHs), and its derivatives can be modified and used in advanced materials. It is also commonly used in paints and optical brightening agents. Furthermore, fluorene is widely used as a ligand (Zou et al., 2007; Han & Wang, 2009). Due to its high donor and acceptor properties, various applicable methods have been developed for the synthesis of fluorene derivatives in organometallic chemistry and various coordination mechanisms (Xu et al., 2015). The literature also mentions the creation of a series of oligofluorenes (OFs) with increasing conjugation lengths, which can be attached at the 2- and 7-positions (Abbel et al., 2009). Fluorene exhibits a planar structure due to the freedom of rotation around the biphenyl bond (Abbel et al., 2009).

According to research by Venkateswararao and colleagues, incorporating the phenyl/fluorene/carbazole unit has allowed for the modification of the conjugation bridge between the donor and acceptor in organic solar cells. Fluorene and carbazole, as electron-rich units, have improved the redox stability of dyes and their dye regeneration tendencies. In this study, they used carbazole compounds as donors in organic solar cells due to their good thermal and electrochemical stability and their hole-transporting properties. They assumed that adding fluorene units would effectively enhance conjugation and serve as an effective charge-delocalizing segment, especially when compared to 3,6-substituted carbazoles. The newly developed dyes outperformed their counterparts with simple phenyl spacer units in terms of optical and electrochemical properties. Experimentally determined values included a V_{oc} value of 634-636 mV and efficiency percentages of 4.20-4.62. The results suggested that the addition of fluorene and carbazole units provided benefits to optical and electrochemical processes, and it was also observed that fluorene units generally increased molar extinction coefficients in absorption (Venkateswararao et al., 2012).

In this study, 3,6-disubstituted carbazole compound containing fluorene was synthesized using bromination, alkylation, and Suzuki-Miyaura cross-coupling reactions. In these reactions, fluorene, known for its high fluorescence and electron-deficient properties at the -3 and -6 positions of carbazole, was used with fluorene boronic acid, enhancing conjugation. The structural characterizations of this synthesized compound were determined using FT-IR, ^{1}H-NMR, ^{13}C-NMR, and elemental analysis, and photophysical and thermal gravimetric analyses were conducted using UV-Vis, FL, TGA-DTA spectra. After elucidating the structure of the synthesized carbazole derivative, its potential use as a dye in TiO_2-based Dye-Sensitized Solar Cells (DSSCs) with a new electron donor-hole system was investigated.

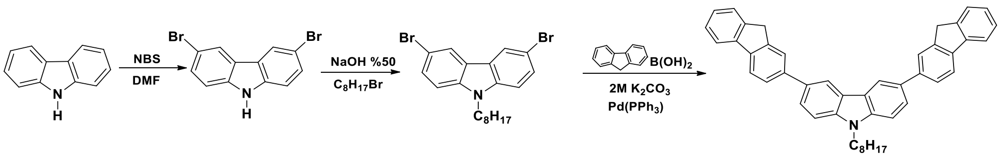
Materials and Method

Instruments and Measurements

All chemicals and solvents used in the reactions were sourced from Sigma Aldrich (Turkey) and abcr. Organic solvents were of HPLC grade or purified using standard procedures. Milli-Q grade water (18.2 MΩ cm) was used for all experiments. FT-IR spectra of all compounds were obtained using a Bruker FT-IR Spectrophotometer (4000-400 cm^{-1}). NMR spectra were recorded in $CDCl_3$ with TMS as the internal standard for ^{1}H-NMR (500 MHz) or an appropriate solvent as the internal standard for ^{13}C-NMR (125 MHz) using a Jeol ECZ500R (11.75 Tesla) NMR instrument. The UV-Vis absorption spectra of the target compound were acquired using a SHIMADZU UV2450 UV-Vis scanning spectrophotometer, while fluorescence spectra were obtained with a Perkin–Elmer LS-55 spectrophotometer. Thermal analyses were conducted using a Perkin Elmer STA 8000 instrument (25°C-1600°C) in the Central Research Laboratory Application and Research Center (ARUM). The current-voltage characteristics of the Dye-Sensitized Solar Cells (DSSCs) were recorded using a FYTRONIX OPTOSENSE solar cell measurement system, with a voltage sweep rate of 0.01V/s under AM1.5G conditions.

Synthesis of Sensitizer IV

The synthesis method and scheme used to achieve the target compound are summarized in Scheme 1



I II III IV

Figure 1. Synthesis scheme of compounds II, III and IV

.Synthesis of 3,6-dibromo-9H-carbazole (II) and Synthesis of 3,6-dibromo-9-octyl-9H-carbazole (III) (Derince et al., 2022). Bromination (II) and alkylation (III) reactions were carried out, as in our previous studies (Derince et al., 2022), to activate the 9H-carbazole and enhance its solubility, and these reactions were characterized.

Synthesis of 3,6-di(9H-fluorene-2-yl)-9-octyl-9H-carbazole (IV)

Reaction of Suzuki Miyaura coupling (Miyaura, 2004) 3,6-dibromo-9-octyl-9H-carbazole (1 eq.) with of fluorene-2-boronic acid (2.2 eq.) catalyzed by 2% $Pd(PPh_3)_4$ and 2M K_2CO_3 (7.5 eq.) was carried out in a mixture of water and 1,2-dimethoxyethane (2:1, DME/H_2O, v/v). Reaction mixture was continued at 80°C for 24h. in N_2 atmosphere. Crude product was washed with DCM and water. It was obtained which the residue was purified by column chromatography (silica gel, petroleum ether: DCM (5:1), affording 3,6-di(9H-fluorene-2-yl)-9-octyl-9H-carbazole (IV) as a yellowish powder. Isolated yield: (75%) ^{1}H-NMR (500 MHz, $CDCl_3$, δ ppm): 8.45 (qz s, 2H), 7.91(s, 2H), 7.87 (d, 2H *J*=7.96 Hz), 7.81 (d, 2H, *J*=7.63Hz), 7.79(dd, 2H, *J*=8.48Hz), 7.76 (dd, 2H, *J*=8.00 Hz), 7.56 (d, 2H, *J*=7.44 Hz), 7.49 (d, 2H, *J*=8.89 Hz), 7.39 (t, 2H *J*=8.16 Hz), 7.30(t, 2H, *J*=8.46 Hz), 4.24 (t, 2H), 4.00 (s, 4H), 1.82 (m, 2H), 1.20-1.30 (m, 10H), 0.85 (t, 3H). ^{13}C NMR (125 MHz, $CDCl_3$, δ ppm): 143.971, 141.610, 140.803, 140.362, 132.644, 126.799, 126.511, 125.407, 125.042, 123.890, 123.545, 120.137, 119.859, 118.947, 109.089, 37.051, 31.820, 29.420, 29.209, 27.366, 22.624, 14.100.

TiO_2-DSSC Fabrication

Commercially available TiO_2 photoanodes (were kept in the dark for 1 day in a chloroform solution containing the new 3,6-disubstituted carbazole compound containing fluorene sensitizer (IV) with a concentration of $1x10^{-3}$ M. It was observed that the photoanodes absorbed the dyes during this 24-hour period. After 24 hours, these electrodes were removed from the solution, washed with ethanol, dried on a 100°C heater, and then covered with Meltonix (a transparent sealing film). Iodine solution [iodide/triiodide (I^-/I^{-3})] was injected onto the films, and they were sealed with a platinum-coated electrode. Thus, the fabrication of Dye-Sensitized Solar Cells (DSSCs) was completed.

Results and Discussion

Absorption and Emission Properties of Synthesized IV

To determine the photophysical properties of the compound synthesized for dye-sensitized solar cells (DSSC), different polarity solvents (THF, DMF, DCM, DMSO, and TOLUEN) were selected and used to measure the maximum absorption and emission wavelengths. In studies conducted with the Fluorene fluorophore, Wang and colleagues (Chen et al., 2022) observed bathochromic shifts in the emission spectra with increasing solvent polarity (TOLUEN < THF < EtOH < H_2O). Compound number IV, which we synthesized, also contains both carbazole and fluorene groups, and it has been observed that bathochromic shifts occur with increasing solvent polarity. The UV-Vis absorption and emission spectra of this compound in various polar solvents are shown in Figure 2(a) and Figure 2(b).

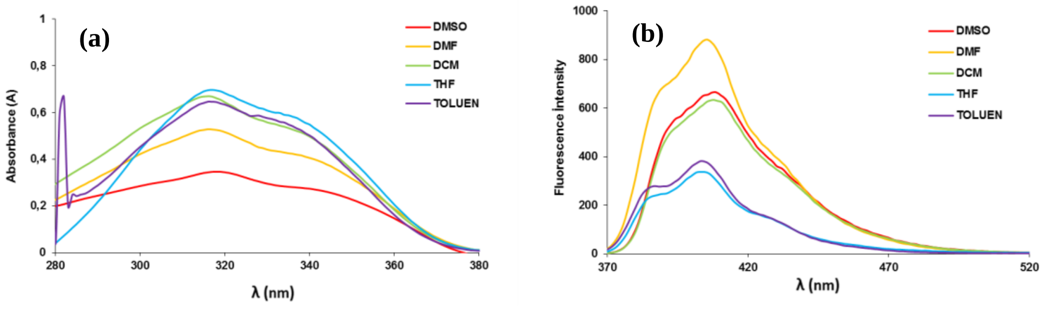


Figure 2. (a) UV-Vis (Ultraviolet-Visible) Spectrum of 3,6-di(9H-fluoren-2-yl)-9-octyl-9H-carbazole (10^{-5} M)
(b) Fluorescence Spectrum of 3,6-di(9H-fluoren-2-yl)-9-octyl-9H-carbazole (10^{-7} M)

When the photophysical properties of the π-conjugated systems were examined based on the UV-Vis absorption spectra, it was observed that the absorption values of the π-π* transitions in the synthesized molecules, influenced by the carbazole groups, varied between 294.0 nm and 306.0 nm (Figure 2(a)). According to the absorption spectra of compound IV in different solvents, the absorptions observed between 316.0 nm in DCM shifted to 318.0 nm in DMSO solvent. This can be explained as a bathochromic shift in the chromophore substitution absorption due to the increase in solvent polarity.

When the emission spectra of compound IV in different solvents were examined, a bathochromic shift was observed at 405.5 and 400.0 nm in DMSO solvent, respectively, while a hypsochromic shift was observed in the emission spectra at 388.5 and 389.0 nm in toluene solvent with decreasing solvent polarity. Additionally, the solvatochromic effect of these compounds was calculated using the Stokes shift value, which demonstrated a bathochromic shift in DMSO solvent (Table 1).

Table 1. Absorption and emission data of compounds IV in various solvents.

Compound	Solvent	$\lambda_{(max)\ abs}$ (nm)	$\lambda_{(max)\ ems}$ (nm)	Stokes shift (cm^{-1} M^{-1})	(cm^{-1} M^{-1})
IV	DMSO	318	408	6937	34634
	DMF	316	405	6954	52869
	DCM	316	408	7135	64987
	THF	317	403	6732	69703
	TOLUEN	316	404	6893	64644

Thermal Stability of Synthesized IV

The thermal stability of the compound with the determined IV number, synthesized in accordance with the target, was investigated using thermal gravimetric analysis (TGA) and differential thermal analysis (DTA), and these data are presented in Figure 3.

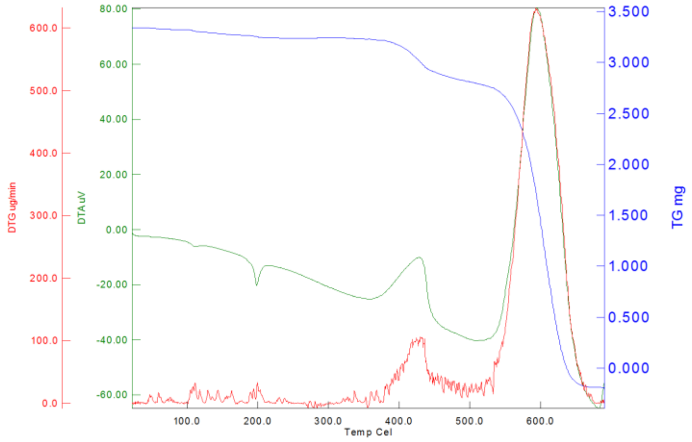


Figure 3. TGA mass loss and DTA plots of compounds IV taken under N_2 atmosphere.

The temperature data for this compound was observed to be 285.72˚C, which corresponds to the temperature of thermal decomposition with a 5% mass loss. Based on the thermal stability data of the synthesized compound IV, it was found to exhibit good thermal stability.

FT-IR Spectra of Synthesized II, III and IV Compounds

All spectra were displayed in Figure 4 for determining the functional groups in the synthesized compounds. In the synthesis of a new conjugated system containing donor and π-spacer groups, a bromination reaction was carried out using *N*-bromosuccinimide with carbazole as the starting material, resulting in the formation of 3,6-dibromocarbazole (II). According to the FT-IR spectrum, the characteristic N-H stretching band of carbazole appeared at 3421 cm^{-1}, the N-H bending signal at 1564 cm^{-1}, and the signal of C-Br groups at 562 cm^{-1}. In the second stage of the reaction, 3,6-dibromo-9-octylcarbazole (III) was synthesized through an alkylation reaction of 3,6-dibromocarbazole (II), and the characteristic N-H stretching signal of carbazole at 3421 cm^{-1} disappeared. Additionally, C-H stretching bands related to the aliphatic group were observed at 2957, 2922, and 2851 cm^{-1}. For the targeted compound, 3,6-bis(3,5-bis(fluorene)phenyl)-9-octyl-9H-carbazole (IV), a Suzuki-Miyaura cross-coupling reaction was performed with palladium catalysis. In the FT-IR spectra of the synthesized compound, the characteristic signal of the C-Br group, as observed in compound III at 562 cm^{-1}, was found to be absent. Furthermore, in the synthesized compound, it was observed that the signals at 1164 cm^{-1} and 797 cm^{-1} belong to the strong bending signals of the 1,4-disubstituted phenyl ring. Additionally, in the synthesized compounds, aromatic C-H stretching vibrations were observed just above 3000 cm^{-1}, while aliphatic C-H stretching vibrations were observed just below 3000 cm^{-1}.

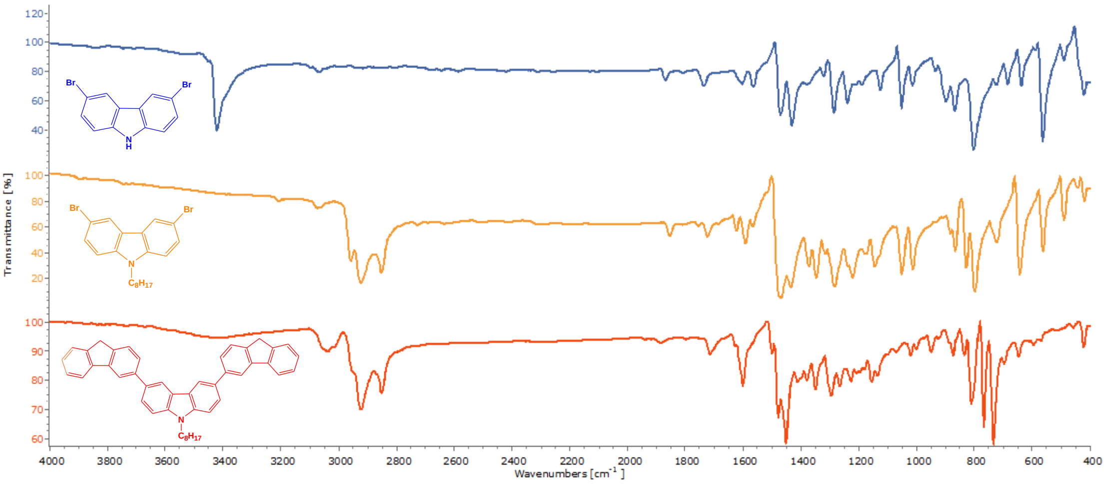


Figure 4. FT-IR spectra of compounds I, III and IV.

^{1}H-NMR Spectra of Synthesized II, III and IV Compounds

The structures of the synthesized compounds II and III were elucidated using ^{1}H-NMR and ^{13}C-NMR spectra obtained in $CDCl_3$, and it was observed that these spectra were in accordance with the literature (Li et al., 2004). The targeted compound IV has a total of 30 protons. The presence of these protons was observed in the ^{1}H-NMR spectrum, and the relevant spectrum is provided in Figure 5.

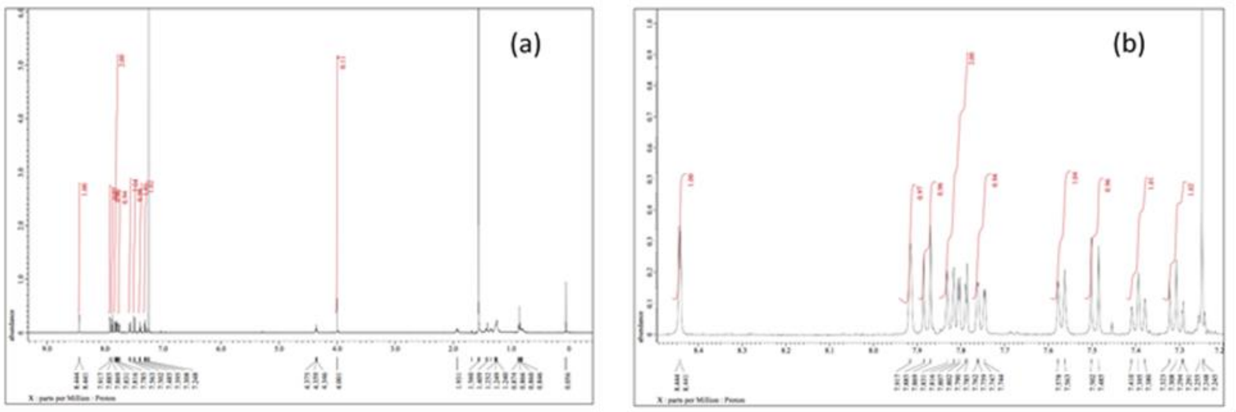


Figure 5. The ^{1}H NMR spectrum of compound IV in $CDCl_3$.

According to the signals observed in this spectrum, it was determined that the aromatic region protons belong to the phenyl rings within the carbazole and fluorene moieties, accounting for a total of 20 protons calculated in the ^{1}H-NMR spectrum. Additionally, in Figure 5, the presence of alkyl groups is indicated by the triplet of two protons at 4.39 ppm, multiple signals of two protons at 1.93 ppm, multiple signals of ten protons at 1.28-1.25 ppm, and triplets of three protons observed at 0.86 ppm, which are attributed to the aliphatic octyl group.

Electrochemical Study

The electrochemical properties of the I, III and IV were investigated using CV using DCM solution containing NBu_4PF_6 as the supporting electrolyte and ferrocene as the internal standard. The obtain CV voltomograms are shown in Figure 7, and corresponding data are summarized in Table 2. The onset oxidation potential was observed in range of 0.998, 1.292 and 0.896 V for I, III and IV, respectively. The HOMO energy levels were calculated using these onset value as negatively 5.58, 5.76 and 5.32 eV for I, III and IV, respectively. Also, the LUMO energy levels of molecules were calculated according to estimate from the onset wavelength of the optical absorption bands (Figure 6). Additionally, the HOMO energy levels of carbazole-derived compounds used in solar cells were determined as -5.67 and -6.02 eV (Agarwal et al., 2011). Therefore, it can be concluded that the HOMO energy level of the carbazole derivative molecule “IV” synthesized in our study, is suitable for use as an active material in solar cells.

Table 2. Optical and electrochemical properties synthesized compounds

	E_{onset}^{ox} [a] (V)	HOMO[b] (eV)	LUMO[c] (eV)	λ_{onset} [d] (nm)	Eg_{opt} [e] (V)
I	0.998	-5.58	-2.46	397	3.12
III	1.292	-5.76	-2.43	372	3.33
IV	0.896	-5.32	-2.09	384	3.23

[a] Determined from cyclic voltammetry experiments vs. Fc/Fc^+ in DCM.

[b] Calculated based on HOMO= - [E_{onset}^{ox} - $E_{1/2,\ Fc,Fc+}$ + 4.8] eV (Sathiyan et al., 2016)

[c] Obtained from LUMO = HOMO + $E_g$opt (Yang et al., 2018)

[d] Calculated from the cross point of absorption onset line and corrected base line.

[e] Estimated from the onset wavelength of the optical absorption bands.

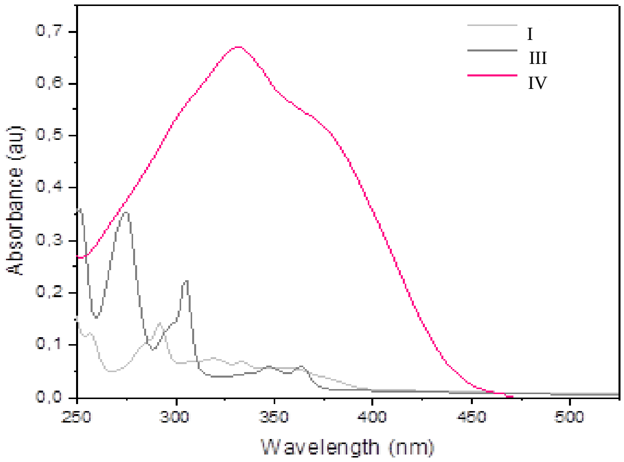


Figure 6. Absorption spectra of compounds I, III, and IV in DCM solution (10^{-5} M)

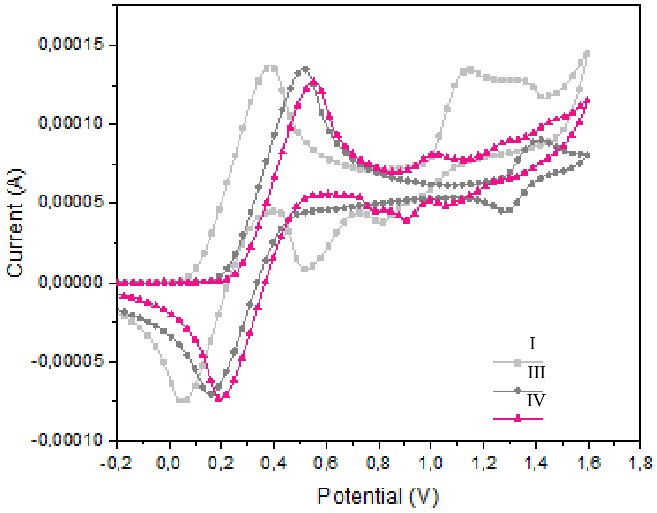


Figure 7. Cyclic voltammograms of compounds II, III and IV under room conditions; Au as working electrode; scan rate ν = 50 mV/s, 0.1 M Bu_4NPF_6 in DCM.

Photovoltaic Properties of TiO_2-DSSC

We were used in the fabrication of dye-sensitized solar cells (DSSC) of compound IV, alongside commercially available TiO_2 nanopowders (TiO_2-DSSC). These DSSCs were constructed on FTO substrates, and electrical measurements were conducted. The performance values obtained are presented in Table 3. Additionally, current density-voltage graphs of the prepared TiO2-DSSC, and the commercially available ones are depicted in Figure 8.

Table 3. The photovoltaic parameters of TiO_2-DSSCs

DSSC	**V_{oc} (V)**	**FF**	**n (%)**
TiO_2-Ru(535)bis TBA	0.631	0.520	0.570
TiO_2-**IV**	0.490	0.370	0.253
TiO_2-**IV**-Ru(535)bis TBA	0.650	0.630	5.572

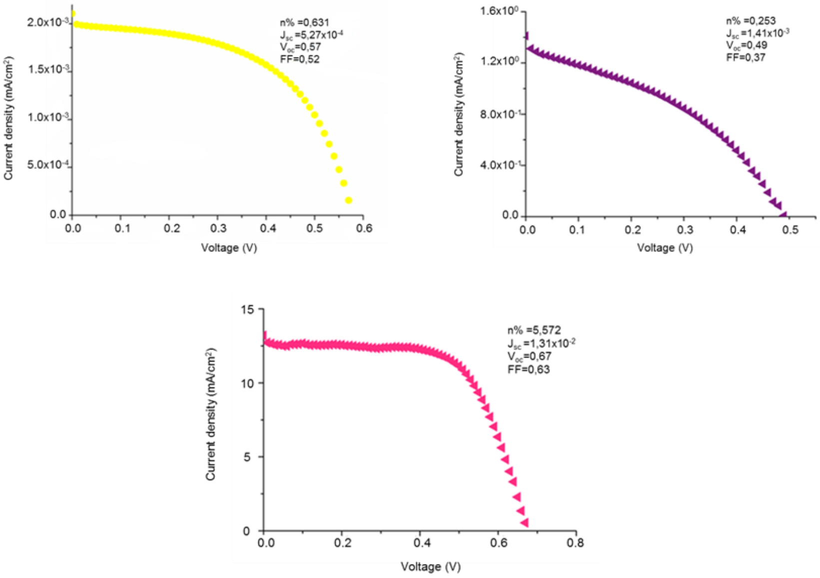


Figure 8. J-V graph of TiO_2-DSSCs

The electrical measurements showed that the power conversion efficiencies (PCE) of the cells fabricated with the prepared using the commercially available TiO_2 nanopowders exhibited power conversion efficiencies ranging from 0.253 to 5.572%, with V_{oc} values varying between 0.370 and 0.630V. In addition, fluorenes are part of intramolecular charge transfer dyes (ICT or push-pull). In these compounds, the π-conjugated systems typically consist of multiple bonds (olefin or acetylene), aromatic rings (such as fluorene or benzenoids), heteroaromatics (e.g., thiophenes), or combinations of these. Besides being categorized as D-π-A systems, there are other arrangements like quadrupolar (D-π-A-π-D) and octupolar ((D-π)3-A) systems. The significant spectroscopic properties and the linear characteristics of the carbazole-fluorene compound (IV) we used as a π-conjugated system may contribute to V_{oc} values ranging from 0.37 to 0.65 and PCE values between 0.253 and 5.572.

Conclusion

In this study, 3,6-dibromo-9-octylcarbazole and aryl boronic acids containing the fluorene group were used to synthesize 3,6-bis(3,5-bis(fluorene)phenyl)-9-octyl-9H-carbazole (IV) through a Suzuki-Miyaura cross-coupling reaction. Prior to the reaction, 3,6-dibromocarbazole (II) and 3,6-dibromo-9-octylcarbazole (III) were synthesized through bromination and alkylation reactions. The synthesized carbazole derivative compounds (II, III, and IV) were characterized using FT-IR, ^{1}H NMR and ^{13}C NMR, analysis techniques. Additionally, the UV-Vis absorption and emission spectra of these compounds were obtained in different solvents (DMSO, DMF, DCM, THF, and TOLUEN). It was observed that the π-π* transitions shifted towards longer wavelengths (red-shifted) with increased conjugation within the carbazole group. Thermal gravimetric analyses were conducted to assess the thermal stability of the synthesized compounds, and the temperature value for 5% mass loss of compound IV was found to be 285.72°C. Dye-sensitized solar cells (DSSC) were fabricated using compound IV on FTO-coated TiO_2. The efficiencies of the prepared TiO_2-DSSC-Ru(535)bis TBA, TiO_2-DSSC-IV, and TiO_2-DSSC-IV-Ru(535)bis TBA dye-sensitized solar cells were found to be in the range of 0.253-5,572. The V_{oc} values were found to be in the range of 0.28-0.63V. The fluorene groups linked through the 3,6- positions of the carbazole unit can lead to the reduction of the hole injection barrier, thereby facilitating the injection of positive charge carriers. The fluorene groups linked through the 3,6- positions of the carbazole unit can lead to the reduction of the hole injection barrier, thereby facilitating the injection of positive charge carriers.

Scientific Ethics Declaration

The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

Acknowledgements or Notes

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