

Design, Synthesis and Studies of Carbazole and Triazine Based Materials for Optoelectronic Applications

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Abstract—Carbazole is a photo and electro luminescent moiety having good thermal stability. Many carbazole based molecules used in organic electronic for fabrication of electronic devices. Three new triazine and carbazole based dyes were prepared through a series of steps involving alkylation, bromination and coupling. All the compounds were characterized through IR, ¹H- and ¹³C-NMR and elemental analysis. Different studies i.e. (photo physical, thermal and electrochemical) were performed in order to check their applicability for electronic devices. The results indicate that compounds emit in the range of blue-green with excellent thermal stability that make their use in devices favorable. Thus, linking of electron donating moieties to triazine improves the characteristic and renders their use feasible for organic electronics applications.

Keywords— *Optoelectronic, thermal, molecular materials, photophysical*

I. INTRODUCTION

Organic based thin-film optoelectronic devices, including light emitting diodes (OLEDs), organic solar cells (OSCs), and organic thin film transistors (OTFTs), preserve great economical potentials; they may direct to a new generation of consumer electronic devices that might be prepared at low cost and beneficial to large areas of life. Conjugated small molecules semiconductors are very significant because of their exceptional optical and electrical characteristics which will allow the production of new optoelectronic devices which have distinctive functions. These conjugated small molecule semiconductors have much improved understanding of the electronic structure, charge-transport properties and charge-charge interaction in related thin films [1-5].

Organic semiconductors which are based on π -conjugated systems are centre of huge significance in the rising area of smooth and elastic photonic and electronics. But in current years the performances of devices like organic light emitting diodes, organic field-effect transistors or solar cells is highly evaluated. Many technical and primary troubles associated with low dimensionality of organic semiconductors due to linear π -conjugated systems still poorly set on [6]. Current transmission through holes is important as well as electron transfer in semiconductors. Densities of thermally produced electrons and holes in semiconductors are normally very small at room temperature gives the thermal energy of 26 meV at room temperature [7]. Host material acts essential function in shaping the device performances for example: external quantum efficiency, current efficiency and duration through

charge stability and competence energy transport. One of the extensively used building blocks for host materials in blue OLEDs is a carbazole moiety which has brilliant hole-transporting property and huge triplet energy. Hole-transporting materials which are based on the carbazole moiety have been focus on the rising amount of the investigation over the last decade. This might be elaborated by very motivating characteristics like low rate of initial material (9H- carbazole), fine chemical and environmental stability given by the totally aromatic unit, simple substitution of the nitrogen atom with a variety of functional groups allowing a better and good modification of the electronic and optical properties [8]. But problems associated with the organic based devices are low life time and stability.

To overcome the limitations, many host materials having bipolar properties have been developed. To arrange bipolarity in the carbazole-based host material structures, a variety of moieties, able to have electron-accepting like pyridine, triazole, triazine, phenanthroline, oxadiazole, benzimidazole, phosphine oxide, and phosphine sulfide were included in a bipolar host material [9, 10].

Herein we report the synthesis and studies of carbazole and triazine based molecular materials by varying degree of terminal groups to enhance the π -conjugation of triazine for better performance and to assess their effect on various properties i.e. optical, thermal, and electrochemical.

II. EXPERIMENTAL

A. Instrumentation

Infrared spectra were recorded using a FTX 3000 MX spectrophotometer using the ATR method. ¹H NMR and ¹³C NMR spectra were obtained using a Bruker AM (300 MHz, 75 MHz) spectrophotometers respectively in CDCl₃, DMSO-d₆ solution using TMS as an internal. Thermogravimetric analysis. Elemental analyses was done using CHNS 932 LECO instrument. (TGA) was performed using a PerkinElmer Thermal analysis System 409. Differential Scanning Calorimetry (DSC) measurements were performed using Bruker Reflex II thermosystem. UV-Vis spectra were recorded using a CECIL-7400 UV/Visible Spectrophotometer and fluorescence spectra were recorded using the Hitachi FL solutions 7000 fluorescence spectrophotometer. Cyclic 0.1M TBAP as supporting electrolyte in CHCl₃ (purged with argon for 10 minutes) on platinum as working electrodes versus Ag/AgCl reference electrode and platinum wire as counter electrode at room temperature.

B. Synthetic Procedures**3, 6-Dibromo-N-octylcarbazole (2)**

Bromine (1.8 mL, 35.7 mmol) was added dropwise to a mixture of N-octylcarbazole (4g, 15mmol) and acetic acid (10 mL). The mixture was stirred for 3 hours at room temperature. The solid product formed were filtered and recrystallized from ethanol giving white crystals.

Yield: 83%; m.p.: 77-78°C; IR (cm^{-1}): 3061 (sp² C-H stretching), 2959, 2854 (sp³ C-H stretching), 1589 (Ar C=C), 1465 (CH₂ bend), 1375 (CH₃ bending), 1319 (C-N7), 673(C-Br).

3-Bromo-9-octylcarbazole (3)

To a mixture of N-octylcarbazole (1g, 3.5 mmol) in chloroform (12 mL) stirring in a covered flask at 0°C was added solution of NBS (0.62g, 3.5 mmol) in CHCl₃ (20 mL). Flask was warmed to room temperature after complete addition, and stirred further for 6 hrs. After completion of reaction the mixture water was added and extraction was carried out. Rotary evaporation provided brown oil which was purified upon column chromatography using hexane giving a colorless oil.

Colourless oil; Yield: 87%; Rf: 0.73; IR (cm^{-1}): 3074 (sp² C-H stretching), 2967, 2829 (sp³ C-H stretching), 1594 (Ar C=C), 1464 (CH₂ bending), 1373 (CH₃ bending), 1321 (C-N), 681 (C-Br).

6-bromo-9-octyl-9H-carbazole-3-carbonitrile (4)

To a mixture of 3,6-Dibromo-N-octyl carbazole (0.8g, 2.23mmol) and DMF (2 ml) was added Copper cyanide (0.2g,2.23mmol). The mixture was heated to 140⁰ in an oil bath for 30 minutes. Then cooled to room temperature and 2ml of cold water was added. Then mixture was stirred at room temperature for 01 hour. The solid material was filtered and transferred to new flask and 2ml water was added. KCN (0.45g, 6-69mmol) was added and mixture was heated to 50⁰ in an oil bath for 01 hour. It was cooled to room temperature; solid was filtered and washed twice with water. Then solid dissolved in 5ml CHCl₃ and dried with anhydrous Na₂SO₄. Solvent was removed under vacuum to afford the product.

White powder; Yield: 69%; Rf: 0.64; IR (cm^{-1}): 3064(sp³ C-H stretch), 2919, 2878(sp² C-H stretch), 2253(C-N stretch), 1594(C=C), 671(C-Br); ¹H-NMR (CDCl₃, 300 MHz): 7.93(d, J=7.8Hz, 1Ar-H), 7.86(d, J=8.1Hz, 1Ar-H), 7.72(d, J=7.4Hz, 1Ar-H), 7.52-7.47(m, 3Ar-H), 4.32(t, J=7.6Hz, 2H, N-CH₂), 1.99-1.96(m, 2H), 1.41-1.29(m, 10H), 0.93(t, J=8.2Hz, 3H, CH₃); ¹³C NMR (CDCl₃, 100 MHz) 141.2, 135.7, 131.9, 128.9, 127.2, 126.4, 125.3, 123.7, 119.8, 112.7, 111.9, 106.4, 103.2, 59.2, 33.4, 30.3, 29.6, 27.8, 23.1, 14.8.

9-octyl-9H-carbazole-3-carbonitrile (5)

To a mixture of 3-Bromo-9-octylcarbazole (1.8g, 4.1mmol) and DMF (8ml) CuCN (0.29g, 3mmol) was added. This mixture was heated for 30 minutes at 140°C in oil bath. Then cooled to room temperature, added 16ml cold water and stirred mix at room temperature for 01 hour. Solid was filtered and transferred to new flask with 16ml of water. KCN (1g) was added and solution was heated to 50°C in oil bath for 01 hour. It was cooled to room temperature; solid was filtered and washed twice with H₂O. Solid dissolved in 10ml CHCl₃ and dried with anhydrous Na₂SO₄. Solvent was removed in rotary to get an oily product.

Oily product; Yield: 81%; Rf: 0.67; IR (cm^{-1}): 3071(sp² C-H stretch), 2932, 2871(sp³ C-H stretch), 2249(CN stretch), 1589(C=C), 1321(C-N); ¹H-NMR (CDCl₃, 300 MHz): 8.22(d, J=8.1Hz, 1Ar-H), 7.96(d, J=1.6Hz, 1Ar-H), 7.67-7.61(m, 2Ar-H), 7.52-7.47(m, 3Ar-H), 4.34(t, J=7.9Hz, 2H, N-CH₂), 1.94-1.89(m, 2H), 1.39-1.27(m, 10H), 0.88(t,

J=6.98Hz, 3H, CH₃); ¹³C NMR (CDCl₃, 100 MHz) 143.2, 131.1, 127.8, 126.3, 125.7, 123.2, 122.8, 121.1, 119.8, 112.6, 110.4, 104.6, 102.4, 58.4, 32.3, 30.6, 29.8, 27.5, 22.9, 14.7.

9-octyl-9H-[3,9'-bicarbazole]-6-carbonitrile (6)

To a mixture 6-Bromo-N-Octylcarbazole-3-carbonitrile (1.21g, 3.15mmol) and DMSO (20ml) was added CuI (0.08g, 0.42mmol) and K₂CO₃ (0.7g, 4.88mmol). To this mixture carbazole (0.6g, 3.59mmol) was added. Mixture was heated to 140-150⁰C in oil bath for 3 days. Upon completion of reaction monitored by TLC it was cooled to room temperature and water was added. The solid material was filtered and extracted with DCM. Organic layer was collected and dried with anhydrous Na₂SO₄. Solvent was removed under vacuo and oily product was obtained

Colorless oil; Yield: 56%; Rf: 0.51; IR (cm^{-1}): 3065(sp³ C-H stretch), 2978, 2832(sp² stretch), 2251(CN stretch), 1324(C-N); ¹H-NMR (CDCl₃, 300 MHz): 8.41 (d, J = 7.7 Hz, 1Ar-H), 8.26 (d, J = 7.3 Hz, 1Ar-H), 8.02 (d, J = 7.6 Hz, 2Ar-H), 7.86 (d, J = 8.0 Hz, 1Ar-H), 7.74-7.63 (m, 3 Ar-H), 7.46-7.38 (m, 4Ar-H), 7.37 (dt, J₁ =1.3 Hz, J₂ =7.8 Hz, 2Ar-H), 4.23 (t, J = 7.4 Hz, 2H, N-CH₂), 1.87-1.83 (m, 2 H), 1.72-1.26 (m, 10 H), 0.85 (t, J = 7.2 Hz, 3H, CH₃); ¹³C-NMR (CDCl₃, 75 MHz): 141.8, 138.6, 133.3, 129.6, 125.7, 123.2, 122.5, 121.6, 120.4, 119.5, 117.7, 112.1, 111.8, 111.0, 110.4, 110.1, 109.6, 102.3, 43.8, 31.7, 30.9, 29.4, 27.6, 22.4, 14.3.

6-(diphenylamino)-9-octyl-9H-carbazole-3-carbonitrile (7)

To a mixture of 6-Bromo-N-Octylcarbazole-3-carbonitrile (0.2g, 0.52mmol) and DMSO (15ml) was added diphenylamine (0.1g, 0.59mmol). Reaction mixture was heated in an oil bath. Then K₂CO₃ (0.25Gg, 1.86mmol) was added. It was cooled to room temperature. Water was added and mixture was extracted with DCM. Lower layer was obtained and dried with Na₂ SO₄ anhydrous. Solid particles were filtered and solvent was removed under rotary to get light brown solid.

Light brown solid; Yield: 61%; Rf: 0.54; IR (cm^{-1}): 3071(sp³ C-H stretch), 2939, 2816(sp² C-H stretch), 2246(CN stretch), 1321(C-N); ¹H-NMR (CDCl₃, 300 MHz): 7.99(d, J=7.9Hz, 1Ar-H), 7.89(d, J=1.4Hz, 1Ar-H), 7.56(dd, J=7.9, 1.4Hz, 1Ar-H), 7.37-7.29(m, 6Ar-H), 7.09(m, 2Ar-H), 6.83(m, 4Ar-H), 6.72-6.69(m, 1Ar-H); ¹³C-NMR (CDCl₃, 75 MHz): 146.8, 137.2, 136.7, 132.2, 129.9, 127.4, 126.1, 125.4, 123.3, 120.1, 114.6, 112.9, 111.8, 109.7, 105.4, 103.7, 55.4, 32.1, 29.8, 29.1, 27.6, 22.3, 14.2.

2,4,6-tris(9-octyl-9H-[3,9'-bicarbazol]-6-yl)-1,3,5-triazine (D1)

To a mixture of 9-octyl-9H-[3,9'-bicarbazole]-6-carbonitrile (1.0g, 2.1mmol) and DCM (20ml) was added F₃CSO₃H (3.2g, 21.3mmol). Reaction mixture was stirred for 30 hours at room temperature. Then it was diluted by CHCl₃. Water was added and mixture was extracted with CHCl₃. Lower layer was obtained and dried with anhydrous Na₂SO₄. Solvent was evaporated under vacuum and brown colour solid was obtained which was purified by column chromatography to afford a pale yellow powder.

Pale yellow powder; Yield: 59%; Rf: 0.61; IR (cm^{-1}): 3074(sp³ C-H stretch), 2938, 2864(sp² C-H stretch), 1596(C=C), 1484(CH₂ bend), 1375(CH₃ bend), 1318(C-N); ¹H-NMR (CDCl₃, 400 MHz): 8.62(d, J=7.9Hz, 3Ar-H), 8.34-8.31(m, 3Ar-H), 8.21(d, J=7.6Hz, 3Ar-H), 7.99-7.91(m, 6Ar-H), 7.81(d, J=8.1Hz, 3Ar-H), 7.72-7.69(m, 6Ar-H), 7.61(m, 3Ar-H), 7.43-7.28(m, 15Ar-H), 4.21(t, J=7.4Hz, 6H, N-CH₂), 1.82-1.79(m, 6H), 1.34-1.28(m, 30H), 0.91(t, J=7.5Hz, 9H, CH₃); ¹³C-NMR (CDCl₃, 100 MHz): 172.4,

142.8, 141.9, 135.4, 131.2, 129.9, 127.3, 124.2, 123.3, 120.7, 117.5, 114.8, 114.3, 113.9, 111.3, 105.2, 55.2, 31.7, 29.6, 28.7, 23.2, 14.7; Anal C₉₉H₉₃N₉: Cal C=84.40, H=6.65, N=8.95, Found C=84.13, H=6.61, N=8.97.

6,6',6''-(1,3,5-triazine-2,4,6-triyl)tris(9-octyl-N,N-diphenyl-9H-carbazol-3-amine) (D2)

To a mixture of 6-(diphenylamino)-9-octyl-9H-carbazole-3-carbonitrile (0.6g, 1.27mmol) and DCM (20ml) was added F₃CSO₃H (1.9g, 12.7mmol). Reaction mixture was stirred overnight at room temperature. Then it was diluted by CHCl₃. Water was added and mixture was extracted with CHCl₃. Upon evaporation under vacuum, brown solid was obtained, purified through column chromatography giving off white solid.

Off white solid; Yield: 62%; Rf: 0.65; IR (/cm⁻¹): 3061(sp³ C-H stretch), 2949, 2872(sp² C-H stretch), 1588(C=C), 1485(CH₂ bend), 1373(CH₃ bend), 1319(C-N); ¹H-NMR (CDCl₃, 400 MHz): 8.36-8.33(m, 3Ar-H), 7.92(d, J=7.7Hz, 3Ar-H), 7.62-7.58(m, 6Ar-H), 7.41-7.32(m, 12Ar-H), 7.09-6.92(m, 12Ar-H), 6.82-6.69(m, 12Ar-H), 4.22(t, J=7.4Hz, 6H, N-CH₂), 1.79-1.74(m, 6H), 1.32-1.27(m, 30H), 0.89(t, J=7.2Hz, 9H, CH₃); ¹³C-NMR (CDCl₃, 100 MHz): 170.2, 146.7, 140.8, 136.7, 132.6, 130.1, 127.7, 125.8, 121.2, 117.1, 114.6, 112.5, 111.3, 109.9, 104.2, 56.4, 31.9, 29.6, 29.0, 28.1, 22.9, 14.5; Anal C₉₉H₉₉N₉: Cal C=84.04, H=7.05, N=8.91, Found C=83.98, H=6.69, N=8.94.

2,4,6-tris(9-octyl-9H-carbazol-3-yl)-1,3,5-triazine (D3)

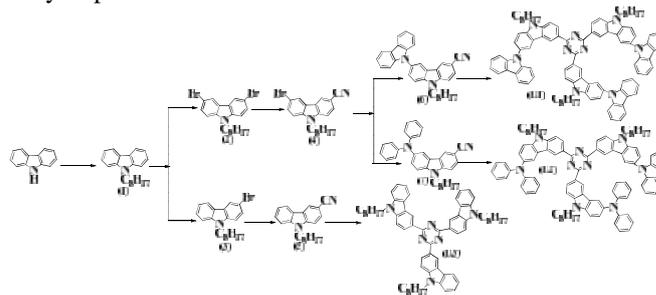
To a mixture of 9-octyl-9H-carbazole-3-carbonitrile (0.5g, 1.64mmol) and DCM (15ml) was added F₃CSO₃H (2.4g, 16.4mmol). Reaction mixture was stirred at room temperature for 20 hours. Water was added and mixture was extracted with CHCl₃. Lower layer was obtained and dried with Na₂SO₄ anhydrous. Solvent was evaporated under vacuum and brown residue was obtained which upon column chromatography provide the white powder

White powder; Yield: 67%; Rf: 0.69; IR (/cm⁻¹): 3041(sp³ C-H stretch), 2916, 2841(sp² C-H stretch), 1583(C=C), 1483(CH₂ bend), 1372(CH₃ bend), 1322(C-N); ¹H-NMR (CDCl₃, 400 MHz): 8.51(d, J=8.0Hz, 3Ar-H), 8.34-8.31(m, 3Ar-H), 7.97-7.88(m, 3Ar-H), 7.83(d, J=7.6Hz, 3Ar-H), 7.71-7.68(m, 2Ar-H), 7.60-7.54(m, 7Ar-H), 4.18(t, J=7.1Hz, 6H, N-CH₂), 1.79-1.77(m, 6H), 1.33-1.29(m, 30H), 0.97(t, J=7.9Hz, 9H, CH₃); ¹³C-NMR (CDCl₃, 100 MHz): 169.8, 139.3, 133.6, 130.7, 123.4, 121.2, 119.8, 119.0, 117.6, 112.8, 110.3, 108.4, 54.1, 31.7, 29.6, 28.4, 26.3, 22.7, 14.5; Anal C₆₃H₇₂N₆: Cal C=82.85, H=7.95, N=9.20, Found C=82.81, H=7.90, N=9.23.

III. RESULTS AND DISCUSSION

Carbazole was alkylated at 9th position by octyl bromide [11], which was further brominated at 3,6 and 3 positions respectively[12,13]. Both mono and di-brominated carbazole were converted to cyano group by direct cyanation with CuCN and KCN [14]. Under the Ullmann coupling conditions the compound (4) was coupled to carbazole and diphenylamine and products were isolated in good yields [15]. In the final step, cyclization of (5, 6, 7) was performed using trifluoromethane sulfonic acid in a simple manner to afford the final products [16]. Effect of changing the donor moiety at the end was checked and assessed. Similarly, effect of alkylated carbazole at terminal position was monitored through cyclization of uncoupled octyl carbazole-3-carbonitrile. Characterization of compounds involves spectroscopic studies and elemental studies. IR spectra show the presence of strong signals for alkyl stretch while NMR

indicates the presence of intense signals in the aromatic region of both proton and carbon. Further confirmation achieved through consistency of calculated and found values of elemental analysis proves the trimerization.



Scheme 1. Synthetic route to dyes D1-D3

A. Photophysical Studies

Ultraviolet-visible spectroscopic analysis was carried out to determine the maximum absorbance (λ_{max}). All dyes absorbed in the visible region between 341-295 nm. The maximum absorbance (λ_{max}) observed due to $\pi \rightarrow \pi^*$ transitions.

All compounds exhibit a secondary band also which is due to $n \rightarrow \pi^*$ transitions and less is in intensity as $n \rightarrow \pi^*$ transitions are forbidden. The presence of high absorption band at 341 nm for D-1 observed due to electron donating carbazole and electron accepting triazine charge transfer interactions. While dye D-2 having diphenylamine group have absorption peak at 333 nm, slightly blue shifted than D-1 possibly due to electronic effect. While D-3 has λ_{max} at 295 nm. This trend observed due to absence of terminally linked electron donating moieties which further enhance the conjugation. The combined spectra are shown in Fig. 1

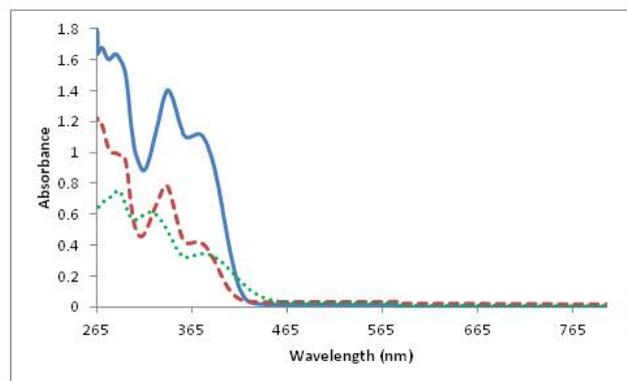


Fig.1 UV/Vis of final dyes D1-3

Emission studies were performed to determine the wavelength of emitted light. Fig. 2 shows the fluorescence studies. All dyes emitted in the visible region between 450-400 nm, and emit light from light blue to yellow-green colors. The dye D2 show maximum emission peak at 450 nm because of presence of terminal diphenyl amine moiety that enhances the path of conjugation while the D1 has $\lambda_{max}(emi)$ at 400 nm, blue shifted of all dyes due to conjugation break appeared by linking carbazole at 3,6 positions. The combined data is provided in Table 1.

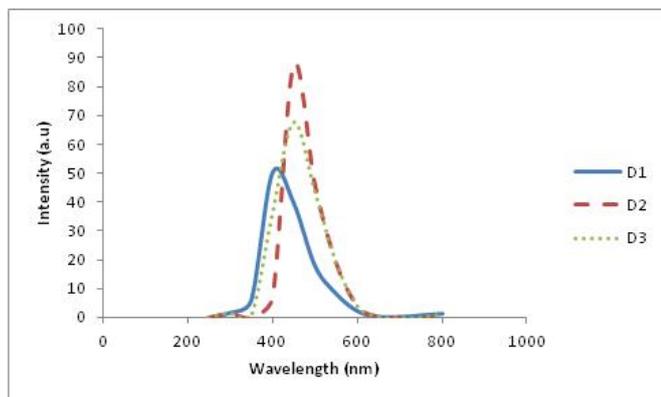


Fig.2 Fluorescence spectra of final dyes D1-3

B. Thermal Studies

Thermal Gravimetric Analysis (TGA) was carried out to check the thermal stability of synthesized compounds. T_d was measured from thermo gram. T_d is the temperature showing the weight loss to the onset of experimental conditions at which changes takes place. All dyes show good thermal stability above 300°C. So, their use in thin-film formation is highly desirable. Thermo grams are shown in Fig. 3.

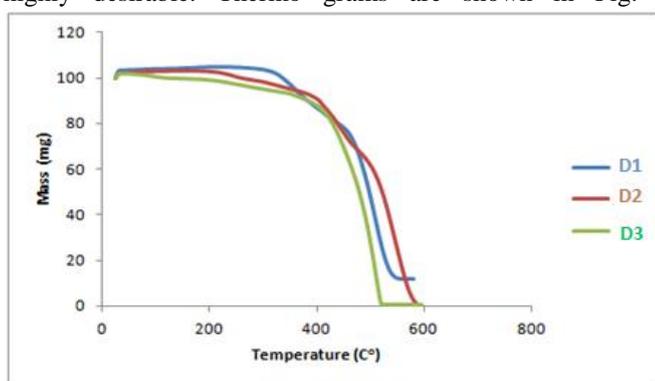


Fig.3 TGA of dyes D1-3

The highest T_d value was observed for D1 due to attachment of 3, 6 linked carbazole unit which provides symmetry whereas D2 shows early decomposition probably due to presence of diphenyl amine flexibility and moisture. DSC measurements were performed in the range of 25-600°C and all the dyes possess the high glass transition temperature (T_g) in the region of 140-146°C. This behavior is indicative of control of amorphous nature of compounds due to donor substituents. Data is given in Table 1.

C. Electrochemical Studies

Cyclic voltammetry studies shows the irreversible oxidation and reversible reduction behavior. From the onset of oxidation HOMO level were calculated lies in the range of -5.44-5.35eV. Fig.4 represents the CV curves of synthesized dyes. The values of HOMO, LUMO and band gap is provided in Table 1.

Table 1: Thermal, optical and electrochemical data of dyes D1-D3

	T_d^a (°C)	T_g^b (°C)	λ_{max}^{abs} (nm) ^c	λ_{max}^{em} (nm) ^d	ϵ^e	HOMO ^f (eV)	LUMO ^g (eV)	Band gap ^h (eV)
D1	394	140	341, 367	400	0.79	-5.35	-2.361	2.990
D2	346	142	333, 374	450	0.78	-5.40	-2.482	2.919
D3	383	146	295, 319	446	0.71	-5.44	-2.709	2.733

^a, ^bThermal decomposition temperature T_d , glass transition temperature T_g determined through TGA and DSC at heating rate of 10°C/min under nitrogen.

^c, ^dDetermined in dilute $CHCl_3$ as 10^{-6} M solution.

^eFluorescence quantum yield was calculated relative to 9,10-Diphenylanthracene ($\Phi_F=0.85$ in $CHCl_3$)

^fHOMO was calculated employing ferrocene value of 4.8 eV below the vacuum level.

^gEstimated from HOMO and band gap by using a relation Band gap (E_{o-o}) = HOMO-LUMO

^hCalculated from the absorption threshold of UV-Visible absorption spectrum

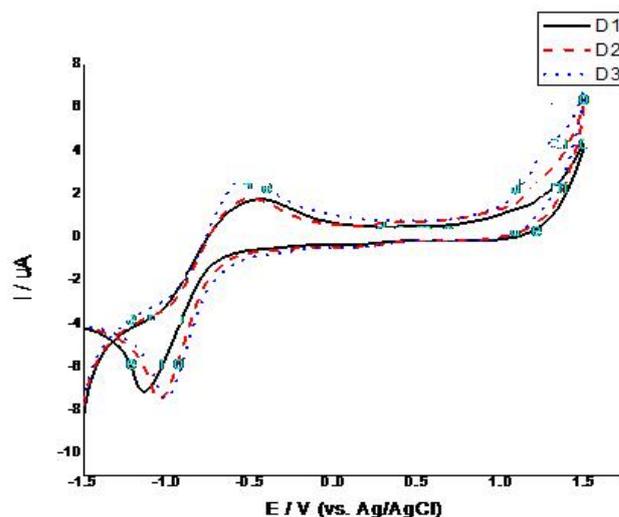


Fig. 4 CV curves of dyes D1-3.

The low lying HOMO value was observed for D1 while the other dyes have HOMO level at 5.40, 5.44 eV. This behavior observed due to electron deficient triazine and different apparently of other carbazolic compounds. Band gap were calculated from HOMO and LUMO values.

CONCLUSION

Three new triazine and carbazole containing molecular materials have been synthesized with changed terminal groups. The effect of altering the terminal groups was studied and it was observed that all dyes possess the appropriate properties associated with combining the donor acceptor moieties and emit in the blue-green region. Dye D1 and D2 has better properties than D3 because of increased donor content. Among the other two, D1 have the improved thermal, electrochemical properties and band gap. Overall, the enhanced thermal, optical and electrochemical properties make these materials to be used in optoelectronic applications.

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