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Synthesis and optical recording properties of some novel styryl dyes for DVD-R

Chung-Chun Lee, Andrew Teh Hu*

Department of Chemical Engineering, National Tsing Hua University, Hsin-Chu, Taiwan 30043, ROC

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Abstract

The synthesis and spectral properties of styryl dyes **6a–6d**, having julolidinyl derivative moieties at one side of the styryl dye structure, are described. These dyes are designed to have different side groups with either carbonate, ether or sulfonate linkages on the julolidinyl ring. Differences in optical, thermal and optical recording properties between these dyes have been compared. The relationships between the side groups and optical/thermal properties of the dyes are discussed.

Keywords: Styryl dye; Julolidinyl group; Electron donor; Side-chain effect; Optical recording; DVD-R

1. Introduction

Styryl dyes are of commercial importance, not only because of their pigment applications, but also due to their application in high value-added products, such as sensitizers in photography [1], fluorescent probes [2], optical recording materials [3,4] and laser dyes [5]. They are chemically stable and also can be tailor-made for a broad range of applications by introducing different side-chains [6,7]. Appropriate substituents can increase the solubility of the dyes and change their optical, thermal or electronic properties to meet the requirements for opto-electronic or biological products [8].

The literature [9–11] shows that the julolidinyl group is a strong electron-donor which can influence the strength and overlap of a nitrogen donor orbital with a conjugated system. Based on this concept, we introduced a julolidinyl ring into a styryl dye to obtain absorption within the range preferred for DVD-R application. In addition, to adjust the thermal properties of the styryl dye, we introduced different side groups in the julolidinyl ring, such as ether, carbonate and sulfonate groups. The optical and thermal performances of the modified products seem to closely meet the preliminary requirements of DVD-R recording materials.

2. Results and discussion

2.1. Synthetic strategy

Using the Vilsmeir-Haak reaction [12], 9-formyl-1, 1, 7, 7-tetramethyl-2, 3, 6, 7-tetrahydro-1*H*,5*H*-pyrido [3,2,1-*ij*] quinoline (compound **3**) was synthesized. From this intermediate, can

* Corresponding author. Tel.: +886-3-571-5131x5704; fax: +886-3-571-5408.
E-mail address: athu@che.nthu.edu.tw (A.T. Hu).

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easily be obtained many kinds of substituted julolidinyl derivatives such as compound **4a–4d** which contains ether, ester and methanesulfonic ester linkages. Furthermore, 1-butyl-2,3,3-trimethyl-4,5-benzo-3*H*-indole iodide (compound **5**) was also synthesized. Using the general Knoevenagel reaction [13], styryl dyes **6a–6d** were obtained from compound **5** and compounds **4a–4d**, respectively. All products were purified using column chromatography and were characterized using IR, ^{1}H NMR and FAB–MS. The route of the synthesis is given in Fig. 1.

2.2. UV–visible absorption spectra of styryl dyes 6a–6d

The UV–visible spectra of **6a–6d** in solution (ethanol/toluene) and thin-film are shown in Figs. 2 and 3, respectively.

In Fig. 2(a), the λ_{max} values of **6a–6d** were as follows: 586 nm for **6a**, 590 nm for **6b**, 550 nm for **6c** and **6d**, from which, it is apparent that dyes **6a** and **6b**, having electron-donating groups (–OR) showed a bathochromic shift. Comparatively, dyes **6c** and **6d** having electron-withdrawing groups (–OCOR and –OSO_2R) displayed a hypsochromic shift. Additionally, the shape of curves **6a** and **6b** are similar but differ to that of curves **6c** and **6d**. Similar results are shown in Fig. 2(b) when toluene was used as solvent instead of ethanol. The change in λ_{max} values for **6c** and **6d** (from 549 to 567 nm) can be attributed to the different polarities of the solvents and the dyes.

In Fig. 3, the λ_{max} values of **6a–6d** thin-films were as follows: 620–570 nm for **6a** and **6b**, 600 nm for **6c** and 575 nm for **6d**. These results, together with comparison to the λ_{max} obtained in solution, suggest that the λ_{max} values of the thin-films had large, red-shifts of about 30 nm.

The above results indicate that the two kinds of side-chain groups used both electron-donating and electron-withdrawing, influence the electronic resonance of the styryl dyes and give rise to two kinds of entirely different UV–visible spectra.

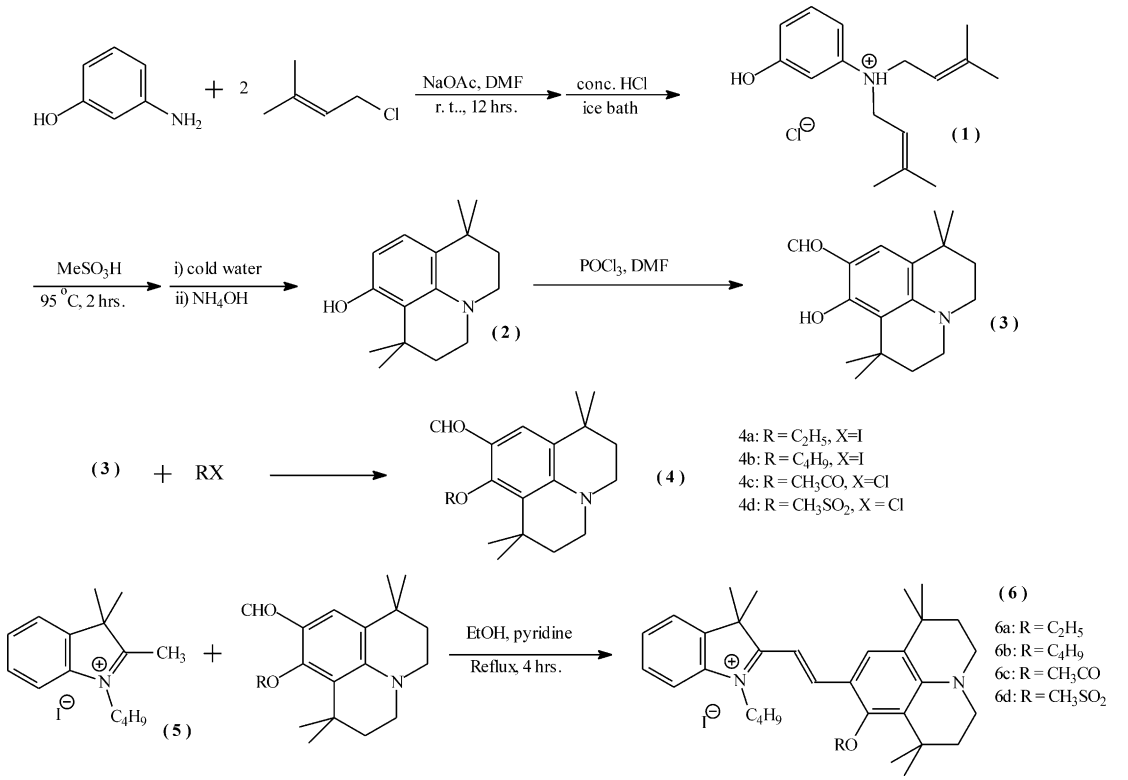

Fig. 1. Synthetic pathway for styryl dyes.

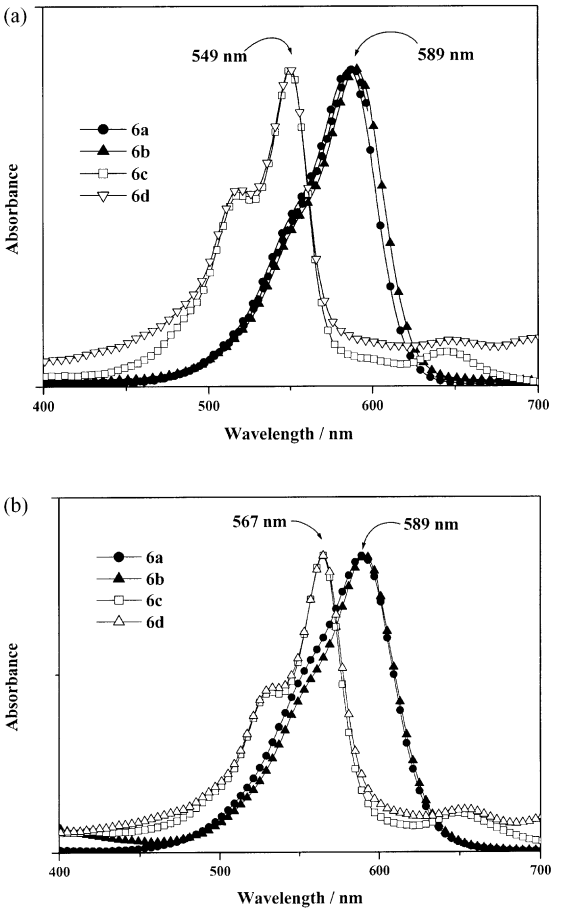

Fig. 2. Absorption spectra of styryl dyes in (a) EtOH and (b) toluene.

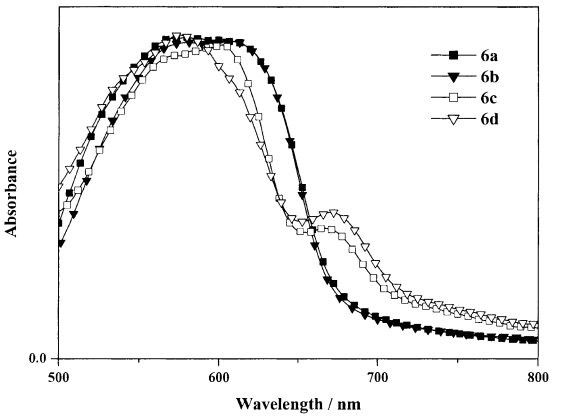

Fig. 3. Absorption spectra of thin-film styryl dyes.

2.3. Thermal properties

The thermal degradation temperatures of dyes **6a–6d** varied for the different side groups as shown in Table 1. Dyes with ether linkages, **6a** and **6b**, decomposed at 210 and 185 °C, respectively. Whilst dyes with carbonate and sulfonate groups, **6c** and **6d**, decomposed at a slower rate and at higher temperatures (236 and 225 °C).

It is postulated that the side groups play an important role in the thermal degradation behavior of the dyes in so far as dyes with ether-linkages degraded at a lower temperature than those with carbonate and sulfonate side-chains.

2.4. Optical recording properties

Fig. 4 shows the reflectance of **6a** before and after silver sputtering on a polycarbonate DVD disc. In addition, Table 1 shows the results of an

Table 1
The thermal degradation temperatures and optical recording testing of styryl dyes

Compound	T_d (°C)	n value[a]	k value[a]	Ref. (%)[b]
6a	207.7	2.380	0.340	35.9
6b	185.6	2.288	0.349	28.6
6c	236.6	1.950	0.200	24.9
6d	225.6	1.877	0.240	39.2

[a] Complex refractive index: $R = n + ik$.

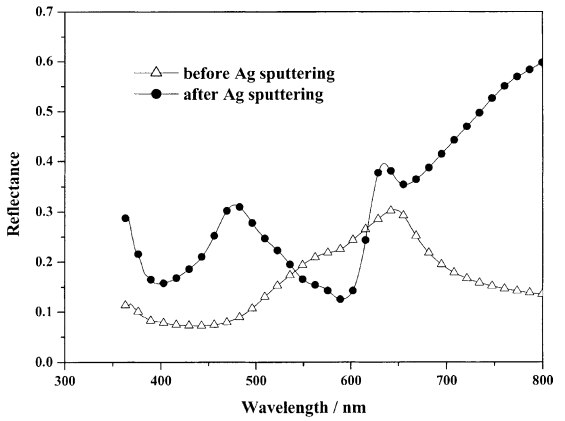

Fig. 4. Reflection spectra of styryl dye **6a** spin-coated on a polycarbonate DVD disc.

optical recording test, which contains complex refractive index values n and k (refractive index, $R = n + ik$, where n is the real part and k is the imaginary part), of thin-films and the reflectance of discs after silver sputtering. All of the styryl dyes closely meet the requirement of refractive index range for DVD-R [14] (optimal range of n, k values were 2.1–2.7 and 0.2–0.4, respectively). Also the refractive index values of **6a** and **6b** were similar, but were different to those obtained for **6c** and **6d**. These phenomena reveal that the refractive index is affected by the side groups of the dyes.

3. Experimental details

3.1. Apparatus

The UV–visible spectra were recorded on a HP8453 ultraviolet spectrophotometer, infrared spectra were recorded on a Perkin-Elmer 842 FT-IR spectrophotometer, photo-luminescent spectra were recorded on a Hitachi model F-2500 fluorescence spectrophotometer and ^{1}H NMR spectra were recorded on a Varian unityinova 500 NMR (500 MHz) spectrometer. Tetramethylsilane (TMS) was used as an internal standard. TGA and DSC experiments were performed by DuPont 2100 and DuPont Model 910, respectively.

The styryl dyes as optical recording materials were dissolved in tri-fluoropropanol (TFP) as a 2.0% (w/w) solution, which was through a 2 μm filter, followed by spin-coating in a rotatory manner over one side of a polycarbonate DVD disc substrate to give a recording layer of thickness of 120 nm. Finally, 100 nm of silver was sputtered as a reflective layer. The optical recording properties (reflectivity and refractive index n and k) were measured using a Steag ETA-Optik spectrometer.

3.2. Reagents

The reagents used for synthesis were of synthetic grade and were used without any purification. The chemicals used for spectroscopic analysis were of analytical reagent grade.

3.3. Synthetic procedure

3.3.1. Preparation of N,N-di-(3-methyl-2-butene)-N-(3-hydroxybenzyl) ammonium chloride (*1*)

In a three-necked flask equipped with a nitrogen inlet, a condenser, and a stirrer, 3-aminophenol (l0.99 g, 0.11 mol), 1-chloro-3-methyl-2-butene (25 g, 0.24 mol), sodium acetate (25 g, 0.3 mol) and 30 ml anhydrous DMF were added. The air in the reaction vessel was replaced by nitrogen and the temperature was maintained at 30 °C with stirring. After about 12 h, the solution was filtered and the filtrate collected. An identical volume of conc. $HCl_{(aq)}$ was added at 0 °C. The residues were filtered, washed with THF several times and then dried in vacuo at 100 °C. Yield was 85%. [FT-IR (KBr disc): 3464, 2960, 2913, 2843, 2690, 2643, 2561, 2339, 1673, 1481, 1406, 1369, 1257, 1226, 984, 910, 760, 693, 581 cm^{-1}. ^{1}H NMR (500 MHz, DMSO-*d*): δ 1.50 (s, 6H), 1.56 (s, 6H), 3.57 (s, 1H), 4.08 (s, 4H), 5.19 (t, 2H), 6.83 (d, lH), 7.14 (s, 2H), 7.23 (t, 1H). *m*/*z*: $(M-Cl)^+ = 247$.]

3.3.2. Preparation of 1,1,7,7- tetramethyl-2,3,6,7-tetrahydro-1H,5H-pyrido [3,2,1-ij] quinoline (*2*)

Compound **1** was added to methanesulfonic acid and heated to 95 °C. After 2 h at this temperature, the mixture was cooled to 0 °C and with vigorously stirring. $NH_4OH_{(aq)}$ was added until pH of 7–8 was achieved; the solution was filtered. Solids were collected and dried in vacuo at 100 °C. Yield was 74%. [FT-IR (KBr disc): 3511, 2925, 2843, 1586, 1486, 1431, 1475, 1300, 1257, 1170, 1227, 1096, 946, 791 cm^{-1}. ^{1}H NMR (500 MHz, $CDC1_3$): δ 1.24 (s, 6H), 1.43 (s, 6H), 1.77 (m, 4H), 3.02 (t, 2H), 3.07 (t, 2h), 4.60 (s, 1H), 6.01 (d, 1H), 6.89 (d, 1H). *m*/*z*: $M^+ = 246$.]

3.3.3. Preparation of 9-formyl-1,1,7,7-tetramethyl-2,3,6,7-tetrahydro-1H,5H-pyrido [3,2,1-ij] quinoline (*3*)

Seven millilitres $POCl_3$ was slowly added to 15 ml anhydrous DMF under nitrogen at 0 °C. The mixture was vigorously stirred for 2 h and then compound **2** dissolved in 10 ml anhydrous DMF was added. After 12 h at room temperature, the solution was added dropwise to ice and then

$NaOAc_{(aq)}$ was added until pH of 7–8 was achieved. After vigorous stirring overnight, the filtrate was collected and recrystallized from ethyl acetate. The product was filtered and then dried in vaccuo at 50 °C. Yield was 92%. [FT-IR (KBr disc): 2942, 2849, 1631, 1516, 1402, 1316, 1230, 1196, 1150, 807, 732, 589, 510 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$): δ 1.24 (s, 6H), 1.42 (s, 6H), 1.71 (m, 4H), 3.20 (t, 2H), 3.29 (t, 2H), 7.02 (s, 1H), 9.37 (s, 1H), 12.19 (s, 1H). m/z: $(M)^+ = 274$.]

3.3.4. Preparation of 9-formyl-10-ethoxy-1,1,7,7-tetramethyl-2,3, 6,7-tetrahydro-1H,5H-pyrido [3,2,1-ij] quinoline (**4a**)

NaH (0.36 g, 15 mmol) and compound **3** (2.73 g, 10 mmol) were slowly added to 15 ml anhydrous DMF under nitrogen. The mixture was vigorously stirred for 2 h and then iodoethane (1.87 g, 12 mmol) was added. After 12 h at room temperature, the solution was extracted with ethyl acetate and water. The organic layer was collected and purified by column chromatography (ethyl acetate:*n*-hexane = 1:5). Yield was 82%. [FT-IR (KBr disc): 2937, 2854, 1654, 1586, 1437, 1313, 1226, 1189, 1145, 1027, 587, 500 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$): 6 l.24 (s, 6H), 1.41 (s, 6H), 1.45 (t, 3H), 1.69 (m, 4H), 3.21 (t, 2H), 3.27 (t, 2H), 3.99 (q, 2H), 7.56 (s, 1H), 9.91 (s, 1H). m/z: $(M)^+ = 302$.]

3.3.5. Preparation of 9-formyl-l 0-but oxy-1,1,7,7-tetramethyl-2,3,6,7-tetrahydro-1H,5H-pyrido [3,2,1-ij] quinoline (**4b**)

NaH (0.36 g, 15 mmol) and compound **3** (2.73 g, 10 mmol) were slowly added to 15 ml anhydrous DMF under nitrogen. The mixture was vigorously stirred for 2 h and then iodobutane (2.21 g, 12 mmol) was added. After 12 h at room temperature, the solution was extracted with ethyl acetate and water. The organic layer was collected and purified by column chromatography (ethyl acetate:*n*-hexane = 1:10). Yield was 79%. [FT-IR (KBr disc): 2971, 2925, 2843, 1660, 1579, 1517, 1474, 1431, 1369, 1319, 1238, 1176, 1152, 1108, 1065, 1015, 506 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$): δ 0.96 (t, 3H), 1.24 (s, 6H), 1.41 (s, 6H), 1.48 (m, 2H), 1.68 (m, 4H), 1.84 (m, 2H), 3.20 (t, 2H), 3.27 (t, 2H), 3.94 (t, 2H), 7.56 (s, 1H), 9.92 (s, 1H). m/z: $(M)^+ = 330$.]

3.3.6. Preparation of 9-formyl-10-acetyl-1,1,7,7-tetramethyl-2,3,6,7-tetrahydro-1H,5H-pyrido [3,2,1-ij] quinoline (**4c**)

Compound **3** (3.5 g, 12.8 mmol) and triethylamine (1.42 g, 14 mmol) were dissolved in 5 ml cold chloroform under nitrogen. Acetyl chloride (1.2 g, 15.3 mmol) was then added to this solution. The mixture was vigorously stirred for 4 h at room temperature and then extracted with ethyl acetate and water. The organic layer was collected and purified by column chromatography (ethyl acetate:*n*-hexane = 1:2). Yield was 76%. [FT-IR (KBr disc): 2948, 2854, 1741, 1629, 1605, 1512, 1462, 1437, 1406, 1369, 1313, 1232, 1195, 1158, 1046, 984, 928, 848, 798, 729, 587, 512 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$): δ 1.23 (s, 6H), 1.42 (s, 6H), 1.70 (m, 4H), 2.18 (s, 3H), 3.20 (t, 2H), 3.30 (t, 2H), 9.36 (s, 1H), 12.20 (s, 1H). m/z: $(M)^+ = 316$.]

3.3.7. Preparation of 9-formyl-10-methanesulfonyl-1,1,7,7-tetramethyl-2,3,6,7-tetrahydro-1H,5H-pyrido [3,2,1-ij] quinoline (**4d**)

Compound **3** (3.5 g, 12.8 mmol) and triethylamine (1.42 g, 14 mmol) were dissolved in **5** ml cold chloroform under nitrogen; methanesulfonic chloride (1.5 g, 13 mmol) was then added to this solution. The mixture was vigorously stirred for 4 h, and then filtered. The filtrate was collected and purified by column chromatography (ethyl acetate:*n*-hexane = 1:5). Yield was 73%. [FT-IR (KBr disc): 2948, 2937, 2854, 1766, 1673, 1636, 1598, 1512, 1362, 1313, 1232, 1195, 1158, 791, 729, 587, 500 cm^{-1}. ^1H NMR (500 MHz, $CDC1_3$): δ 1.24 (s, 6H), 1.42 (s, 6H), 1.72 (m, 4H), 2.95 (s, 3H), 3.21 (t, 2H), 3.30 (t, 2H), 9.37 (s, 1H), 12.21 (s, 1H). m/z: $(M)^+ = 352$.]

3.3.8. Preparation of 1-butyl-2,3,3 trimethyl-4,5-benzo-3H-indole iodide (**5**)

Iodobutane (2.2 g, 12 mmol), pyridine (0.86 g, 11 mmol) and 2,3,3-trimethyl indolenium (1.59 g, 10 mmol) were added to 10 ml methyl ethyl ketone. The mixture was vigorously stirred for 6 hours under reflux and then recrystallized from methyl ethyl ketone. Upon cooling, the precipitate

was filtered and washed with acetone to obtain white crystalline solids. Yield was 82%. [FT-IR (KBr disc): 2971, 2937, 1561, 1468, 1431, 767, 689 cm^{-1}. ^1H NMR (500 MHz, $CDC1_3$): δ 0.96 (t, 3H), 1.46 (m, 2H), 1.62(s, 6H), 1.89 (m, 2H), 3.09 (s, 3H), 4.64 (t, 2H), 7.20–7.65 (m, 4H). *m*/*z*: $(M-I)^+ = 216$.]

3.3.9. Preparation of substituted julolidinyl styryl dye (*6a*)

Compound **5** (3.43 g, 0.01 mol) and pyridine (1.185 g, 0.015 mol) were added to 8 g of propylene glycol monomethyl ether, then compound **4a** (3.01 g, 0.01 mol) was added dropwise. The reaction mixture was under reflux and stirred overnight. Upon cooling, the precipitate was filtered and washed with cold heptane to give **6a** (5.07 g) as a greenish solid. Finally, the product was purified by column chromatography (methanol:ethyl acetate:*n*-hexane = 1:1:2). Yield was 75%. [FT-IR (KBr disc): 2971, 2937, 2854, 1586, 1561, 1468, 1437, 1313, 1226, 1189, 1145, 1027, 767, 587, 500 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$): δ 1.01 (t, 3H), 1.34 (s, 6H), 1.47 (s, 6H), 1.55 (m, 5H), 1.78 (m, 2H), 1.83 (s, 6H), 1.92 (m, 2H), 2.82 (t, 2H), 3.56 (t, 2H), 3.63 (t, 2H), 4.05(q, 2H), 4.56 (t, 2H), 7.20–8.40 (m, 7H). *m*/*z*: $(M-I)^+ = 500$. UV(ethanol): $\lambda_{abs} = 586$ nm (log $\varepsilon = 5.07$). PL(ethanol): $\lambda_{em} = 612$ nm (excited at 550 nm).]

3.3.10. Preparation of substitutedjulolidinyl styryl dye (*6b*)

Compound **5** (3.43 g, 0.01 mol) and pyridine (1.185 g, 0.015 mol) were added to 8 g of propylene glycol monomethyl ether, then compound **4b** (3.29 g, 0.01 mol) was added dropwise. The reaction mixture was under reflux and stirred overnight. Upon cooling, the precipitate was filtered and washed with cold heptane to give **6b** (5.62 g) as a greenish solid. Finally, the product was purified by column chromatography (methanol:ethyl acetate:*n*-hexane = 1:1:3). Yield was 71%. [FT-IR (KBr disc): 2974, 2925, 2843, 1579, 1517, 1474, 1431, 1369, 1319, 1238, 1176, 1152, 1108, 1065, 1015, 767, 689, 506 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$): δ 1.01 (m, 6H), 1.35 (s, 6H), 1.47 (s, 6H), 1.56 (m, 6H), 1.78 (m, 2H), 1.84 (s, 6H), 1.92 (m, 2H), 2.83 (t, 2H), 3.56 (t, 2H), 3.63 (t, 2H), 4.00 (q, 2H), 4.55 (t, 2H), 7.20–~8.40 (m, 7H). *m*/*z*: $(M-I)^+ = 528$. UV(ethanol): $\lambda_{abs} = 590$ nm (log $\varepsilon = 5.13$). PL(ethanol): $\lambda_{em} = 609$ nm (excited at 550 nm).]

3.3.11. Preparation of substituted julolidinyl styryl dye (*6c*)

Compound **5** (3.43 g, 0.01 mol) and pyridine (1.185 g, 0.015 mol) were added to 8 g of propylene glycol monomethyl ether, then compound **4c** (3.15 g, 0.01 mol) was added dropwise. The reaction mixture was under reflux and stirred overnight. Upon cooling, the precipitate was filtered and washed with cold heptane to give **6c** (4.86 g) as a greenish solid. Finally, the product was purified by column chromatography (methanol:ethyl acetate = 1:5). Yield was 66%. [FT-IR (KBr disc): 2970, 2948, 2937, 2854, 1741, 1561, 1512, 1462, 1437, 1406, 1369, 1313, 1232, 1195, 1158, 1046, 984, 928, 848, 798, 729, 689, 587, 512 cm^{-1}. ^1H NMR (500 MHz, $CDC1_3$): δ 1.01 (t, 3H), 1.34 (s, 6H), 1.47 (s, 6H), 1.55 (m, 2H), 1.78 (m, 2H), 1.83 (s, 6H), 1.92 (m, 2H), 2.58 (s, 3H), 2.82 (t, 2H), 3.56 (t, 2H), 3.63 (t, 2H), 4.56 (t, 2H), 7.20–8.40 (m, 7H). *m*/*z*: $(M\text{-}I)^+ = 514$. UV(ethanol): $\lambda_{abs} = 550$ nm (log $\varepsilon = 4.53$). PL(ethanol): $\lambda_{em} = 566$ nm (excited at 550 nm).]

3.3.12. Preparation of substituted julolidinyl styryl dye (*6d*)

Compound **5** (3.43 g, 0.01 mol) and pyridine (1.185 g, 0.015 mol) were added to 8 g of propylene glycol monomethyl ether, then compound **4d** (3.51 g, 0.01 mol) was added dropwise. The reaction mixture was under reflux and stirred overnight. Upon cooling, the precipitate was filtered and washed with cold heptane to give **6d** (4.87 g) as a greenish solid. Finally, the product was purified by column chromatography (methanol:ethyl acetate = 1:5). Yield was 74%. [FT-IR (KBr disc): 2973, 2948, 2937, 2854, 1766, 1598, 1512, 1468, 1431, 1362, 1313, 1232, 1195, 1158, 791, 767, 729, 689, 587, 500 cm^{-1}. ^1H NMR (500 MHz, $CDC1_3$): δ 1.01 (t, 3H), 1.34 (s, 6H), 1.47 (s, 6H), 1.55 (m, 2H), 1.78 (m, 2H), 1.83 (s, 6H), 1.92 (m, 2H), 2.82 (t, 2H), 3.09 (s, 3H), 3.56 (t, 2H), 3.63 (t, 2H), 4.56 (t, 2H), 7.20–8.40 (m, 7H). *m*/*z*: $(M\text{-}I)^+ = 549$. UV(ethanol): $\lambda_{abs} = 550$ nm (log $\varepsilon = 4.78$). PL-(ethanol): $\lambda_{em} = 563$ nm (excited at 550 nm).]

4. Conclusions

Novel styryl dyes with julolidinyl derivative moieties (**6a–6d**) were prepared and characterized. Their optical and thermal properties varied according to the different side groups such as ether, carbonate and sulfonate groups. Some of their thermal and optical recording properties (such as reflectance and refractive index values) closely meet the requirements for DVD-R application.

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