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Synthesis and photolysis of r-1,c-2,t-3,t-4-1,3-di(4-R-phenyl)-2,4-bis[2-(5-phenyloxazolyl)]cyclobutane

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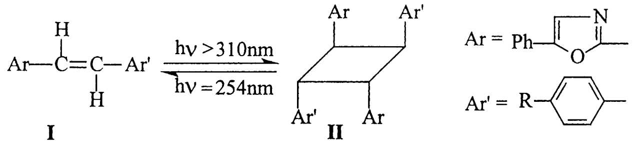
Abstract

r-1,c-2,t-3,t-4-1,3-di(4-R-phenyl)-2,4-bis[2-(5-phenyloxazolyl)]cyclobutane (**IIa**: R = H; **IIb**: R = Cl; **IIc**: R = OMe; **IId**: R = *t*-Bu) was synthesized with high stereo-selectivity via the photodimerization of its corresponding *trans*-monomer in hydrochloric acid. The structures of the title compounds were identified by elementary analyses, IR, UV, ^{1}H NMR, ^{13}C NMR and MS. It has been proved that the photodimers have syn-head-to-tail configuration. The investigation revealed that under irradiation of 254 nm UV light, these photodimers underwent photolysis promptly to reproduce the *trans*-monomers and then the formed *trans*-monomers transformed into their *cis*-isomers by *trans*–*cis* isomerization. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Photodimerization; Photolysis; Cyclobutane derivatives; Oxazole

1. Introduction

Photodimerization of stilbazolium salts [1–3] have been extensively investigated. Under irradiation, these compounds will result in centrosymmetric head-to-tail dimers as the major products. Recently, we reported that some stilbazole derivatives [4–6] and some benzoxazole substituted ethenes [7] can easily undergo photodimerization to form centrosymmetric dimers in water with the presence of mineral acid. Nevertheless, no paper concerns the investigation of the photodimerization of oxazolylethenes and the photolysis of the related photodimers. In this paper, we report our research on the photodimerization of *trans*-1-(4-R-phenyl)-2-[2-(5-phenyloxazolyl)]ethene (**Ia**: R = H; **Ib**: R = Cl; **Ic**: R = OMe, **Id**: R = *t*-Bu) and the photolysis of the formed dimer.



a: R=H; **b**: R=Cl; **c**: R=OMe, **d**: R=t-Bu

2. Experimental details

2.1. Apparatus

Melting points were taken on a Yanagimoto MP-500 apparatus (uncorrected). Elementary analyses were determined on a Yanaco CHNCORDER MF-3 apparatus. The IR and UV spectra were recorded on a BIO-RAD FTS 3000 Infrared and on a HP 8453 UV–VIS spectrophotometer, respectively. The ^{1}H and ^{13}C NMR spectra were taken with a Bruker AC-P200 instrument (^{13}C NMR of **Id**, **IIb** and **IIc** were performed on Varian UNITY-plus 400) using TMS as internal standard. The mass spectra were performed by a VG ZAB-HS spectrometer.

2.2. Preparation of trans-1-(4-R-phenyl)-2-[2-(5-phenyloxazolyl)]ethene

A mixture of 0.01 mol of 2-methyl-5-phenyloxazole [8], 35 ml of DMF, 1.5 g of KOH and 0.015 mol of 4-substituted benzaldehyde was heated to 40 °C and reacted for 6 h under magnetic stirring, followed by treatment of 50 ml of water. The reaction mixture was allowed to stand overnight, the precipitate was collected by filtration and recrystallized from ethanol to give yellow solid.

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Ia (R = H): yield 50%, mp 103–103.5 °C (lit. mp 102–103 °C) [9]. IR (KBr): 1520 (s), 972 (s), 754 (s), 688 (s) cm^{-1}. UV (1,4-dioxane): λ_{max} 338 ($\log\varepsilon$ 4.50) nm.

Ib (R = Cl): yield 73%, mp 148–149.5 °C (lit. mp 148–150 °C) [9]. IR (KBr): 1635 (w), 1522 (m), 1092 (s), 972 (s), 818 (s), 756 (s), 688 (s) cm^{-1}. UV (1,4-dioxane): λ_{max} 342 ($\log\varepsilon$ 4.54) nm. ^{1}H NMR (CDCl$_3$): δ 7.72–7.67 (2H, m), 7.55–7.26 (9H, m), 6.95 (1H, d, J=16.6 Hz); ^{13}C NMR (CDCl$_3$): δ 160.71 (C), 150.99 (C), 134.83 (C), 134.30 (CH), 134.01 (C), 129.08 (CH), 128.92 (CH), 128.52 (CH), 128.26 (CH), 127.75 (C), 124.17 (CH), 123.73 (CH), 114.34 (CH).

Ic (R = OMe): yield 49%, mp 101.5–103 °C. IR (KBr): 1634 (m), 1606 (s), 1028 (s), 1256 (s), 970 (s), 806 (s), 764 (s), 690 (s) cm^{-1}. UV (1,4-dioxane): λ_{max} 348 ($\log\varepsilon$ 4.55) nm. ^{1}H NMR (CDCl$_3$): δ 7.72–7.32 (9H, m), 6.95–6.81 (3H, m), 3.84 (3H, s); ^{13}C NMR (CDCl$_3$): δ 160.43 (C), 150.60 (C), 135.43 (CH), 128.86 (CH), 128.59 (CH), 128.37 (C), 128.26 (CH), 127.97 (C), 124.06 (CH), 123.55 (CH), 114.29 (CH), 111.61 (CH), 108.43 (C), 55.33 (CH$_3$). MS (70 eV, EI): m/z (%) 277 (M^+, 88), 276 ($M - \mathrm{H}^+$, 100), 145 (22). Anal. Calcd. for $\mathrm{C_{18}H_{15}NO_2}$: C, 77.96; H, 5.45; N, 5.05. Found: C, 78.08; H, 5.20; N, 4.97.

Id (R = t-Bu): yield 30%, mp 88–90 °C. IR (KBr): 1642 (m), 1524 (s), 1122 (s), 968 (s), 820 (s), 758 (s), 688 (s) cm^{-1}. UV (1,4-dioxane): λ_{max} 340 ($\log\varepsilon$ 4.53) nm. ^{1}H NMR (CDCl$_3$): δ 7.79–7.36 (11H, m), 7.05 (1H, s), 1.34 (9H, s); ^{13}C NMR (CDCl$_3$): δ 161.53 (C), 152.78 (C), 150.96 (C), 135.92 (CH), 133.04 (CH), 129.11 (CH), 128.58 (CH), 128.20 (C), 127.19 (CH), 126.06 (CH), 124.38 (CH), 123.86 (C), 113.30 (CH), 34.94 (C), 31.41 (CH$_3$). MS (70 eV, EI): m/z (%) 303 (M^+, 73), 302 ($M - \mathrm{H}^+$, 100), 288 ($M - \mathrm{Me}^+$, 24), 246 ($M - \mathrm{Bu}^+$, 6). Anal. Calcd. for $\mathrm{C_{21}H_{21}NO}$: C, 83.13; H, 6.98; N, 4.62. Found: C, 82.89; H, 6.85; N, 4.28.

2.3. Preparation of r-1,c-2,t-3,t-4-1,3-di(4-R-phenyl)-2,4-bis[2-(5-phenyloxazolyl)]cyclobutane

In a 500 ml flask, 2 mmol of **Ia** was dissolved in 200 ml of 20% hydrochloric acid via heating and irradiated with a 300 W medium-pressure mercury lamp (MPML) which was cooled by flowing water through a two-layer glass sheath ($\lambda_{\mathrm{max}} > 310$ nm, the light below 310 nm was absorbed by the sheath). The irradiation was continued until the yellow color disappeared. The reaction mixture was put in refrigerator overnight, the precipitate was filtered and recrystallized from ethanol.

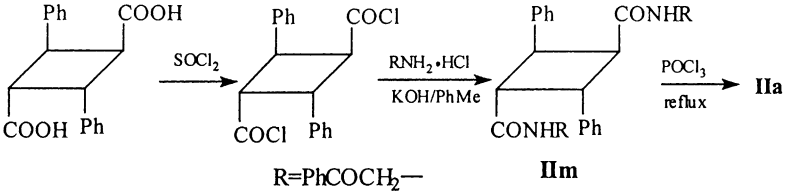
IIa (R = H): **Ia** irradiated for 4 h, yield 82%, mp 222–223 °C. IR (KBr): 1554 (s), 1124 (s), 760 (s), 691 (s) cm^{-1}. UV (1,4-dioxane): λ_{max} 282 ($\log\varepsilon$ 4.62) nm. ^{1}H NMR (CDCl$_3$): δ 7.43–7.14 (20H, m), 7.10 (2H, s), 4.90–4.75 (4H, m); ^{13}C NMR (CDCl$_3$): δ 162.60 (C), 151.46 (C), 138.31 (C), 128.72 (CH), 128.35 (CH), 128.13 (CH), 127.93 (C), 127.24 (CH), 126.97 (CH), 124.04 (CH), 121.67 (CH), 44.72 (CH), 41.26 (CH). MS (70 eV, EI): m/z (%) 247 (**Ia**$^+$, 53), 246 (**Ia** $- \mathrm{H}^+$, 100); MS (100 eV, CI): m/z (%) 523 ($M + \mathrm{Et}^+$, 7), 495 ($M + \mathrm{H}^+$, 25), 276 (**Ia** $+ \mathrm{Et}^+$, 16) 248 (**Ia** $+ \mathrm{H}^+$, 100), 247 (**Ia**$^+$, 86), 246 (**Ia** $- \mathrm{H}^+$, 51). Anal. Calcd. for $\mathrm{C_{34}H_{26}N_2O_2}$: C, 82.57; H, 5.30; N, 5.66. Found: C, 82.68; H, 5.61; N, 5.45.

IIb (R = Cl): **Ib** irradiated for 6 h, yield 73%, mp 208–209.5 °C. IR (KBr): 1552 (s), 1494 (s), 822 (s), 758 (s), 692 (s) cm^{-1}. UV (1,4-dioxane): λ_{max} 283 ($\log\varepsilon$ 4.63) nm. ^{1}H NMR (CDCl$_3$): δ 7.42–7.21 (18H, m), 7.12 (2H, s), 4.82–4.68 (4H, m); ^{13}C NMR (CDCl$_3$): δ 162.22 (C), 152.04 (C), 136.88 (C), 133.20 (C), 129.08 (CH), 128.89 (CH), 128.82 (CH), 128.65 (CH), 127.93 (C), 124.31 (CH), 121.93 (CH), 44.42 (CH), 41.52 (CH). MS (70 eV, EI): m/z (%) 283 (**Ib**(Cl^{37})$^+$, 31), 282 (**Ib**(Cl^{37}) $- \mathrm{H}^+$, 48), 281 (**Ib**(Cl^{35})$^+$, 90), 280 (**Ib**(Cl^{35}) $- \mathrm{H}^+$, 100), 246 (**Ib** $- \mathrm{Cl}^+$, 8); MS (100 eV, CI): m/z (%) 592 ($M + 2 + \mathrm{Et}^+$, 2), 590 ($M + \mathrm{Et}^+$, 3), 566 ($M + 4^+$, 1), 565 ($M + 2 + \mathrm{H}^+$, 2), 564 ($M + 2^+$, 8), 563 ($M + \mathrm{H}^+$, 3), 562 (M$^+$, 11), 284 (**Ib**(Cl^{37}) $+ \mathrm{H}^+$, 25), 283 (**Ib**(Cl^{37})$^+$, 46), 282 (**Ib**(Cl^{35}) $+ \mathrm{H}^+$, 84), 281 (**Ib**(Cl^{35})$^+$, 100), 280 (**Ib**(Cl^{35}) $- \mathrm{H}^+$, 55), 246 (**Ib** $- \mathrm{Cl}^+$, 21). Anal. Calcd. for $\mathrm{C_{34}H_{24}Cl_2N_2O_2}$: C, 72.47; H, 4.29; N, 4.97. Found: C, 72.34; H, 4.36; N, 4.85.

IIc (R = OMe): **Ic** irradiated for 24 h, yield 49%, mp 178–180 °C. IR (KBr): 1614 (s), 1516 (s), 1254 (s), 820 (s), 760 (s), 692 (s) cm^{-1}. UV (1,4-dioxane): λ_{max} 283 ($\log\varepsilon$ 4.64) nm. ^{1}H NMR (CDCl$_3$): δ 7.46–7.21 (14H, m), 7.11 (2H, s), 6.75 (4H, dd), 4.82–4.64 (4H, m), 3.67 (6H, s); ^{13}C NMR (CDCl$_3$): δ 163.06 (C), 158.71 (C), 151.69 (C), 130.74 (C), 128.96 (CH), 128.65 (CH), 128.36 (CH), 128.28 (C), 124.30 (CH), 122.00 (CH), 114.02 (CH), 55.37 (CH$_3$), 44.33 (CH), 42.05 (CH). MS (70 eV, EI): m/z (%) 277 (**Ic**$^+$, 95), 276 (**Ic** $- \mathrm{H}^+$, 100), 246 (**Ic** $- \mathrm{MeO}^+$, 5); MS (100 eV, CI): m/z (%) 555 ($M + \mathrm{H}^+$, 8), 278 (**Ic** $+ \mathrm{H}^+$, 100), 277 (**Ic**$^+$, 83), 276 (**Ic** $- \mathrm{H}^+$, 36). Anal. Calcd. for $\mathrm{C_{36}H_{30}N_2O_4}$: C, 77.96; H, 5.45; N, 5.05. Found: C, 77.85; H, 5.12; N, 5.10.

IId (R = t-Bu): **Id** irradiated for 6 h, yield 63%, mp 214–216 °C. IR (KBr): 2960 (s), 1550 (s), 824 (s), 760 (s), 688 (s) cm^{-1}. UV (1,4-dioxane): λ_{max} 282 ($\log\varepsilon$ 4.60) nm. ^{1}H NMR (CDCl$_3$): δ 7.34–7.23 (18H, m), 7.09 (2H, s), 4.82–4.72 (4H, m), 1.15 (18H, s). ^{13}C NMR (CDCl$_3$): δ 162.75 (C), 151.46 (C), 149.72 (C), 135.32 (C), 128.61 (CH), 128.04 (CH), 128.00 (C), 126.95 (CH), 125.20 (CH), 124.08 (CH), 121.58 (CH), 44.44 (CH), 41.33 (CH), 34.29 (C), 31.14 (CH$_3$). MS (70 eV, EI): m/z (%) 303 (**Id**$^+$, 95), 302 (**Id** $- \mathrm{H}^+$, 100), 288 (**Id** $- \mathrm{Me}^+$, 30), 246 (**Ib** $- \mathrm{Bu}^+$, 9); MS (100 eV, CI): m/z (%) 635 ($M + \mathrm{Et}^+$, 1), 607 ($M + \mathrm{H}^+$, 7), 332 (**Id** $+ \mathrm{Et}^+$, 12) 304 (**Id** $+ \mathrm{H}^+$, 92), 303 (**Id**$^+$, 100), 302 (**Id** $- \mathrm{H}^+$, 94), 288 (**Id** $- \mathrm{Me}^+$, 23). Anal. Calcd. for $\mathrm{C_{42}H_{42}N_2O_2}$: C, 83.13; H, 6.98; N, 4.61. Found: C, 83.34; H, 7.09; N, 4.54.

2.4. Synthesis of **IIa** from truxillic acid



A mixture of 1 g of truxillic acid and 10 ml of $SOCl_2$ was heated to reflux for 3 h, then 50 ml of anhydrous toluene was added and the mixture was distilled until about 10 ml of residue liquid was left.

In a 100 ml of four necked flask, 2.0 g of 2-amino-1-phenylethanone hydrochloride and 30 ml of H_2O was added under stirring, the water solution was cooled to 0–5 °C, then the above toluene solution of truxillyl dichloride and a solution of 2.1 g of KOH in 10 ml of water was added dropwise together within 0.5 h under vigorously stirring and the pH value was maintained at 6–8. After that, the reaction mixture was stirred for another 0.5 h and filtered. The filter cake was washed with water and small amount of ethanol, and recrystallized from DMF to give 1.2 g of colorless solid of diamide **IIm**. Yield 67%, mp 251–253 °C. IR (KBr): 3390 (m), 1686 (s), 1655 (s), 1507 (s), 760 (m), 690 (m) cm^{-1}. ^1H NMR (DMSO-d_6): δ 8.31 (2H, t), 7.89 (4H, d), 7.63 (2H, t), 7.50 (4H, t), 7.28–7.16 (10H, m), 4.49 (2H, dd), 4.23 (4H, m), 4.39 (2H, m).

1.0 g of **IIm** was refluxed with 10 ml of $POCl_3$ for 3 h and then hydrolyzed with 100 ml ice-water, the solid was filtered, washed with water, recrystallized from ethanol to give 0.8 g of colorless product, yield: 86%. Its melting point, IR and NMR spectra are absolutely in agreement with that of **IIa**.

2.5. Photodimerization

0.01 mol/l of **Ia** in ethanol was irradiated under a 20 W low-pressure mercury lamp (LPML, $\lambda = 254$ nm) for 2 min or under MPML for 100 min; 0.01 mol/l of **Ia** in 20% HCl was also irradiated under MPML. These irradiated solutions were detected by HPLC (Varian 5060 apparatus, ODS 4.6 mm × 300 mm, 4:1of MeOH–H_2O, detecting $\lambda = 254$ nm) and the results were shown in Fig. 1.

2.6. Photolysis

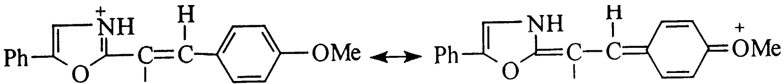
Solutions containing 1.6×10^{-5} mol/l of **Ic** and 0.8×10^{-5} mol/l of **IIc** in 4:1 methanol–water were put in a quartz colorimetric cell and irradiated under LPML for certain time. The reaction mixture was traced by HPLC and UV (Figs. 4 and 5).

3. Results and discussion

3.1. Photodimerization of **Ia–Id** in hydrochloric acid

Irradiated under the MPML, **Ia–Id** in HCl solution produces colorless product. Only one main product for each was isolated. The ^1H NMR, ^{13}C NMR and UV spectra of **IIa–IId** clearly show that they are the photodimers of their corresponding monomers.

It was found that the electron-donating group decreases the photodimerization speed and yield. For example, the photodimerization of **Ia** and **Ib** will complete within 4 and 6 h with yields of 82 and 73%, respectively, while **Ic** needs 24 h to reach its end with a relatively low yield of 49%. It was assumed that in acidic solution, the oxazole ring will be protonated, and the electron-donating group will make the following resonance structure (**IcH**) more stable which is disadvantageous to the photodimerization of **Ic**. So the stronger the electron donating substituted group is, the harder the photodimerization will become.



IcH

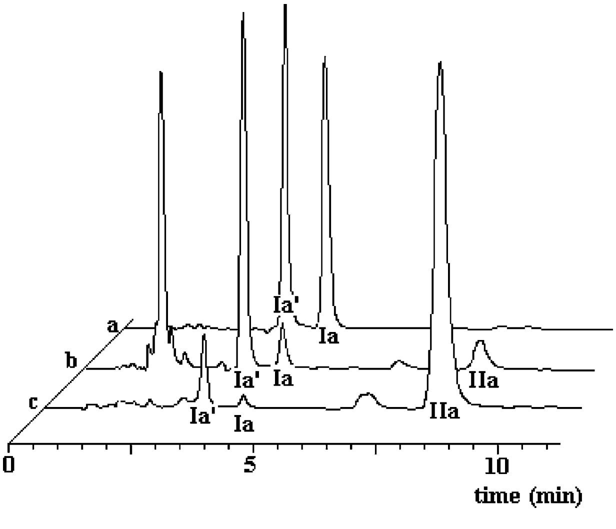
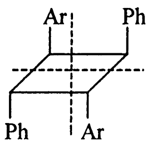


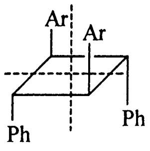
Fig. 1. HPLC traces of 0.01 mol/l of **Ia**. Irradiated under: (a) LPML in ethanol for 2 min; (b) MPML in ethanol and (c) in 20% HCl for 100 min.

3.2. The stereo-structures of the photodimers

Because of the angle strain of the four-membered ring, molecules of **IIa–IId** with high internal energy disintegrate easily at high temperature or impact by electron. For this reason, in the electron impact (EI) mass spectra, their molecular ion peaks are too low to be detected unless they were performed under chemical ionization (CI) condition. There are four possible stereo-isomers. In most cases, the photodimer has syn-head-to-tail structure [2–7,10]. The MS spectra take a further step to demonstrate that. For example, the base peak in the MS spectrum of **IIa** is the molecular ion of **Ia**. Neither the molecular ion of 1,2-diphenylethene (DPE), nor that of 1,2-bis[2-(5-phenyloxazolyl)]ethene (BPE) was detected, which indicates that **IIa** was neither r-1,c-2,t-3,t-4-1,2-diphenyl-3,4-bis[2-(5-phenyloxazolyl)] cyclobutane (**3**) nor r-1,t-2,c-3,t-4-diphenyl-3,4-bis[2-(5-phenyloxazolyl)]cyclobutane (**4**). Because the above two compounds will form $m/z = 152$ and $m/z = 314$ fragment peaks corresponding to the radical cations of DPE and BPE.



1



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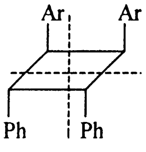

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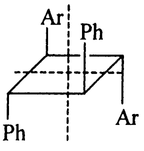

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It was suggested that only in the formation of **1**, an excimer which can decline the activition energy of the dimerization might be existed, so **1** with a syn-head-to-tail structure is the most reasonable structure just like that of those dimers formed from other diarylethenes [2–7].

In order to identify the real structure of these photodimers, **IIa** was also synthesized from truxillic acid, which inevitably demonstrates that **IIa** has syn-head-to-tail structure. By comparing the NMR and MS spectra of the other three compounds with that of **IIa**, It is confirmed that all of these photodimers possess similar syn-head-to-tail structure.

3.3. Photodimerization mechanism

When the ethanol solution of **Ia** was irradiated under LPML, only two peaks (standing for **Ia** and its *cis*-isomer **Ia**′, respectively) were detected (Fig. 1, trace **a**), which means that under irradiation of 254 nm UV light, the main photoreaction of **Ia** is *trans–cis* isomerization; while under MPML, besides the *cis*-isomer **Ia**′, several other products formed slowly, including the identified **IIa** (Fig. 1, trace **b**). However, when the 20% HCl solution of **Ia** was irradiated, **IIa** formed rapidly as a major product and the reaction almost finished after 100 min (Fig. 1, trace **c**). Fig. 1 also proved the photodimerization is faster and more selective in HCl solution than that in ethanol. The maximums of the UV spectra of **Ia** in ethanol and in acetonitrile are 337 and 336 nm, respectively while in 20% HCl solution, it moves to long wavelength region with maximum at 354 nm (Fig. 2). The basic nitrogen atom of the oxazole ring was supposed to be protonated in acidic solvent, which enhances the stability of the formed syn-head-to-tail excimer. So the mineral acids were tested as the reaction media and it was found that **Ia–Id** has suitable solubility in hydrochloric acid.

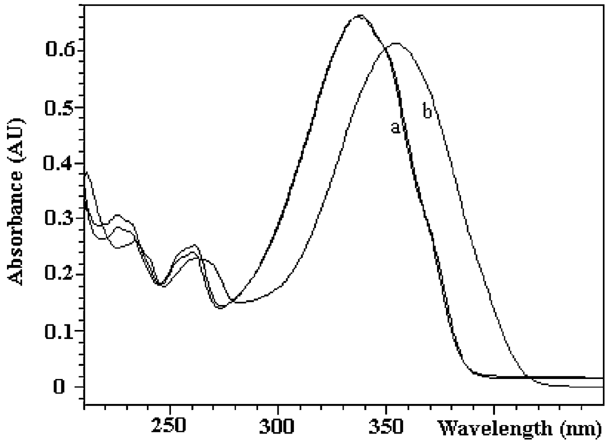

Fig. 2. UV spectra of 2×10^{-5} mol/l of **Ia** (a) in ethanol and acetonitrile, or (b) in 20% HCl.

In HCl solution, **Ia–Id** produces high yield of dimer with high selectivity, and the fact that the photodimerization does not affected by oxygen, which support the excited singlet mechanism [11]. Namely, one excited molecular and one molecular in ground state form an excimer, and then the formed excimer dimerized. The basicity [12] of nitrogen atom of oxazole ring increases at the excited state, in HCl solution, the excited molecule has a protonated oxazole ring and a head-to-tail excimer is more stable than a head-to-head one because of the dipolar attraction between the positively charged oxazole ring of excited molecule and the electron rich benzene ring of the unexcited one.

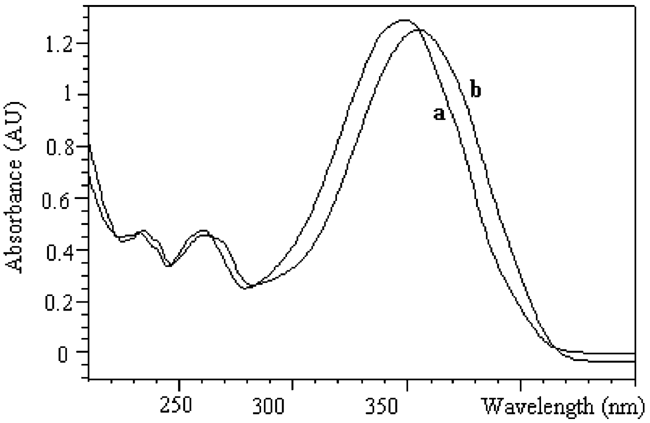

Fig. 3. UV spectra of: (a) 4×10^{-5} and (b) 0.01 mol/l of **Ic** in 20% HCl.

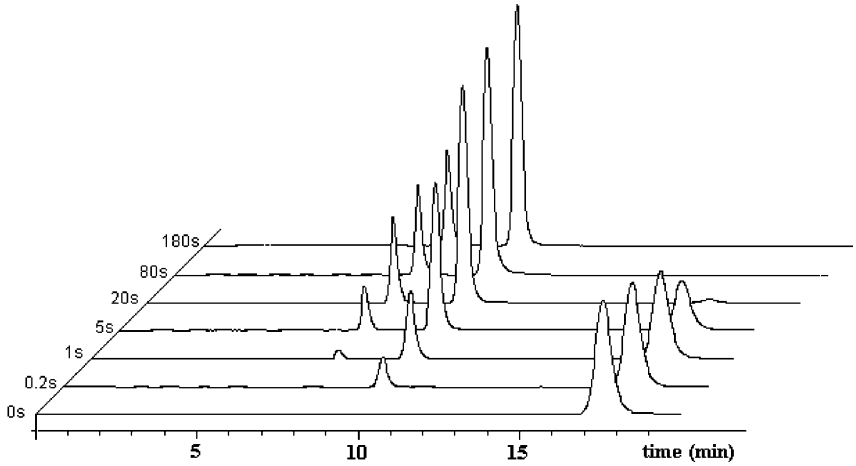

Fig. 4. HPLC traces of **IIc** irradiated under LPML for 0, 0.2, 1, 5, 20, 80, 180 s, respectively.

Ground state aggregation are common phenomena for conjugated aromatic molecules [13–15], usually the absorption spectra of aggregated molecules present blue shift to the short wavelength region. The UV spectra of 4×10^{-5} and 0.01 mol/l (by thin film measurement) of **Ic** in 20% HCl were measured. From Fig. 3, the shapes of the UV spetra are quite similar to each other except that a 3 nm red shift was found for the 0.01 mol/l solution because of the concentration effect. No evidence was detected to demonstrate the existence of the ground state aggregation in this dimerization system.

3.4. *The photolysis of* **IIc** *in 1,4-dioxane*

The photolysis of tetraarylcyclobutane derivatives which produce mixtures of *trans*- and *cis*-diarylethenes have been reported [5,6,16]. However, whether the *cis*-isomer formed directly from the dimer or from the formed *trans*-isomer indirectly was not quite sure. The photolysis of **IIc** was traced by HPLC and UV. At the beginning (irradiation time: 0.2 s, see Fig. 4) of the photolysis, only **Ic** was detected (mid peak). With the irradiation proceeding, the peak of **IIc** (right peak) disappeared gradually, and the peak of **Ic** as-well-as that of **Ic**′ (*cis*-1-(4-methoxyphenyl)-2-[2-(5-phenyloxazolyl)]ethene, left peak) grow up rapidly. When the irradiation time reached to 80 s, all of **IIc** was consumed. Then the *trans–cis* isomerization reached equilibrium, and the end ratio of the peak area of **Ic** to **Ic**′ is 76.9:23.1. It was also found that if the monomer of **Ic** was irradiated for the same time, a similar *tans–cis* mixture was generated. From Fig. 5, the new appeared 348 nm UV peak also implies the photolysis of **IIc** and the formation of its corresponding monomer. The above experimental results proved that the *trans*-monomer formed directly from **IIc** and the *cis*-isomer was formed from the new produced *trans*-isomer through the *trans–cis* isomerization.

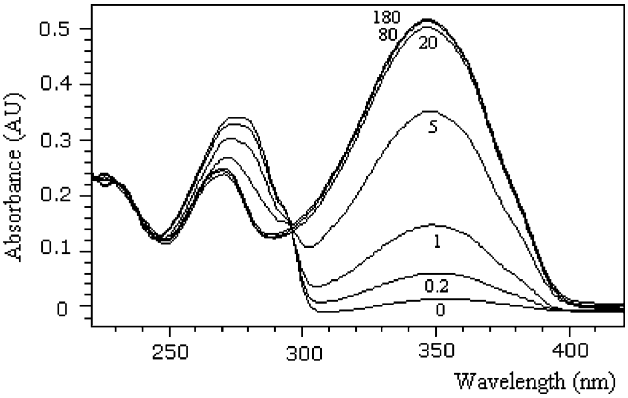

Fig. 5. UV spectra of **IIc** irradiated under LPML for 0, 0.2, 1, 5, 20, 80, 180 s, respectively.

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