

 **Processing '22**

# ZBORNİK RADOVA

**35. Međunarodni kongres  
o procesnoj industriji**

**Holiday Inn, Beograd**

**1–3. jun 2022.**



**SET**  
SAMIT ENERGETIKE TREBINJE



# ZBORNİK RADOVA

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pisanih za 35. Međunarodni kongres o procesnoj industriji  
PROCESING '22



2022

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**pisanih za 35. Međunarodni kongres o procesnoj industriji**  
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Savez mašinskih i elektrotehničkih  
inženjera i tehničara Srbije (SMEITS)  
Društvo za procesnu tehniku  
Kneza Miloša 7a/II,  
11000 Beograd

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# PREDGOVOR

*Od preko 50 radova prijavljenih za ovogodišnji Procesing, za izlaganje je prihvaćeno 47 radova autora iz zemlje i inostranstva.*

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*Međunarodni karakter Procesinga '22 i ove godine ostvaren je inostranim učesnicima sa radovima, kao i članovima naučnog odbora. Zvanični jezici za izlaganje radova na kongresu su srpski i engleski.*

*Osnovni ciljevi kongresa su inoviranje i proširivanje znanja inženjera u procesnoj industriji, energetici, rudarstvu, komunalnom sektoru (vodovodima, toplanama) i podrška istraživačima u predstavljanju ostvarenih rezultata istraživačkih projekata.*

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*Procesing '22 organizuje Društvo za procesnu tehniku pri SMEITS-u, a u Naučnom i Organizacionom odboru prisutni su predstavnici svih Mašinskih fakulteta u Srbiji kao i Tehnoloških i drugih fakulteta u okviru kojih je oblast procesne tehnike zastupljena u nastavi.*

*Pomoć u organizovanju Procesinga '22 dali su članovi Katedre za procesnu tehniku Mašinskog fakulteta Univerziteta u Beogradu i mnogih drugih fakulteta iz Srbije.*

*Ovogodišnji skup završava se posetom novom Centru za upravljanje otpadom u Vinči.*

*U Beogradu  
juni 2022.*



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### **Oglasni deo**

# PROUČAVANJE $\alpha$ -CIJANOSTILBENA KAO POTENCIJALNIH MOLEKULSKIH PREKIDAČA METODOM LINEARNE KORELACIJE ENERGIJE SOLVATACIJE

## A LSER ANALYSIS OF $\alpha$ -CYANOSTILBENS AS POTENTIAL MOLECULAR PHOTOSWITCHES

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*Fotohromni organski molekuli koji se mogu reverzibilno prevoditi iz jednog stabilnog stanja u drugo pod dejstvom svetlosti uveliko se primenjuju u proizvodnji raznovrsnih materijala za primenu u proizvodnji različitih optičkih i elektronskih uređaja. To su najčešće mali organski molekuli koji u okviru svoje hemijske strukture sadrže konjugovane aromatične prstenove i koji podležu brzom *cis*→*trans* fotoizomerizaciji, fotociklizaciji ili oba procesa. Njihov fotohemijski potencijal nije još uvek dovoljno istražen, budući da je teško balansirati efekte geometrijskih i elektronskih promena na molekulskom nivou kako bi se proizvela određena makroskopska svojstva materijala. U cilju dizajniranja novih fotosenzitivnih materijala, u ovom radu predstavljena je solvatrohromna analiza dva 4-supstituisana  $\alpha$ -cijanostilbena i detaljno analiziran uticaj dipolarnosti/polarizabilnosti rastvarača kao i vodoničnog vezivanja na pomeranje apsorpcionog maksimuma primenom metoda linearne korelacije energije solvatacije koji su razvili Kamlet, Taft i Katalan.*

**Ključne reči:** fotohromni molekuli;  $\alpha$ -cijanostilbeni; solvatrohromizam.

*Photochromic organic molecules that undergo reversible photochemical switching between two stable states continue to impact optical devices. They usually represent small organic molecules bearing conjugated aromatic frameworks capable for rapid *cis*→*trans* photoisomerization, photocyclization or combination of both. Their photochromic potential has not been completely studied due to numerous challenges in coupling the geometrical and electronic changes on the molecular level and further balancing macroscopic and bulk material characteristics. With the aim of development of novel light-sensitive materials, herein we explore the solvatochromic behaviour of two 4-substituted  $\alpha$ -cyanostilbens by recording their absorption spectra in selected solvents and evaluating the effects of solvent dipolarity/polarizability and solute-solvent hydrogen bonding interactions on the shift of the absorption maxima by means of linear solvation energy relationship concepts proposed by Kamlet, Taft and Cátalan.*

**Key words:** photochromic molecules;  $\alpha$ -cyanostilbens; solvatochromism

## 1 Introduction

According to literature overview, photochromism is the feature of some organic compounds to undertake a light-induced reversible change of color based on a chemical reaction [1]. As extrinsic mild trigger, light has been broadly employed [2] for sophisticated regulation at the supramolecular and macroscopic level—from light-triggered nanomaterials to photocontrol over biological systems [3].  $\pi$ -Conjugated organic compounds, especially, stilbene and its derivatives, which can regulate its outstanding features in response to a variety of physical or chemical factors, have become an indispensable part of structural architectures such as polymers, functional nanoparticles, solid membranes and supramolecular systems. They are essential components for modern optical and photoelectronic industrial production, with special reference to the fabrication of OLED (Organic Light-Emitting Diode), OFET (Organic Field Effect Transistor), chemosensors, bioimaging detectors and medicinal

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treatment products. Since the quantum yield for stilbene is relatively low and its emission band is mostly located in shortwave spectral region, the structural modification and chemical optimization of this photochromic compound has been intensively applied. Overall, these modifications of the stilbene scaffold led to construction of cyanostilbene, a kind of derivative molecules of stilbene composed of a well-stretched  $\pi$ -conjugated molecular plane with a covalently linked –CN-group in the *para* position of the aromatic ring. The presence of the cyano group and other side groups extend the  $\pi$ -electron cloud distribution of stilbene making the conjugation system broader which further results in more efficiently light emission and covering of the visible region in the spectrum [4]. In the first part of this work, the synthesis and structural characterization of two 4-substituted  $\alpha$ -cyanostilbens (Figure 1), as potential molecular photoswitches, will be described, while the second part is focused on their solvatochromic properties interpreted using linear solvation energy relationship concept proposed by Kamlet, Taft and Catalán.

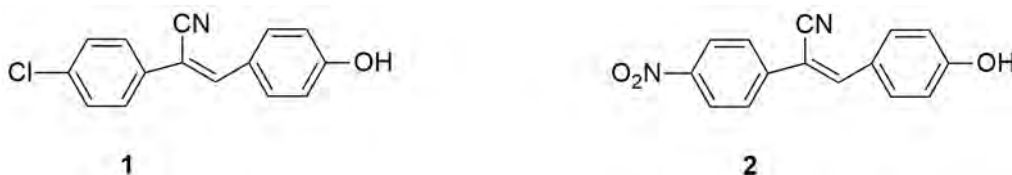
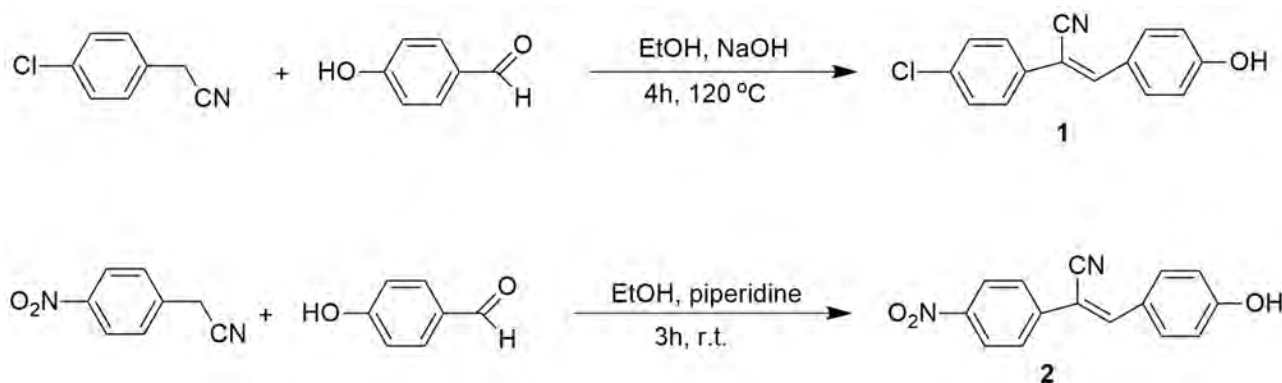


Figure 1. Chemical structure of the investigated compounds

## 2 Experimental

### 2.1 General

Chemicals used in the synthesis of the investigated compounds were obtained from Sigma Aldrich chemical company and were used without further purification. Solvents used were of spectroscopic grade.  $\alpha$ -Cyanostilbens (**1,2**) were prepared according modified literature procedure [5,6] applying synthetic pathway illustrated on Scheme 1. The IR spectra were recorded using a Bomem MB-Series Fourier Transformer-Infrared (FT-IR) spectrophotometer in the form of KBr pellets. The ultraviolet-visible (UV-Vis) absorption spectra were recorded on a Shimadzu 1700 spectrophotometer in the region 200–500 nm. The spectra were run in spectroquality solvents (Fluka) using a concentration of  $1 \times 10^{-5}$  mol dm<sup>-3</sup>. All melting points were determined on Electrothermal apparatus.



Scheme 1. Synthesis of the investigated compounds

### 2.2 Synthesis of (Z)-2-(4-Chlorophenyl)-3-(4-hydroxyphenyl)acrylonitrile (**1**)

4-Chlorobenzylcyanide (3.03 g, 20 mmol) and 4-hydroxybenzaldehyde (2.0 g, 17 mmol) were dissolved in absolute EtOH (20 ml). Then NaOH (1.4 g, 36 mmol) was added and the reaction mixture was stirred at reflux for 4h. The mixture was subsequently cooled to room temperature and acidified with HCl. The crude product was collected and recrystallized from ethyl acetate/chloroform to obtain the product [5].

**(Z)-2-(4-Chlorophenyl)-3-(4-hydroxyphenyl)acrylonitrile (**1**):** Brown crystalline solid; Yield: 45%; m.p. 209–212 °C; FT-IR/ATR: (KBr)  $\nu$  (cm<sup>-1</sup>): 2230, 1612, 1518, 1449, 1286, 1177, 1089; <sup>1</sup>HNMR (400 MHz, DMSO-d<sub>6</sub>,  $\delta$ /ppm): 7.88 (s, 1H, -CH), 7.84 (d, 2H,  $J = 8.8$  Hz, Ar-H), 7.70



(d, 2H,  $J = 8.8$  Hz, Ar-H), 7.52 (d, 2H,  $J = 8.4$  Hz, Ar-H), 6.90 (d, 2H,  $J = 8.4$  Hz, Ar-H);  $^{13}\text{C}$  NMR (100 MHz, DMSO- $d_6$ ,  $\delta/\text{ppm}$ ): 157.8, 147.6, 133.8, 132.0, 130.9, 128.9, 127.8, 118.6, 115.7, 99.5.

### 2.3 Synthesis of (Z)-3-(4-Hydroxyphenyl)-2-(4-nitrophenyl)acrylonitrile (2)

To a mixture of the 4-hydroxybenzaldehyde (2.0 g, 16.39 mmol) and 4-nitrobenzylcyanide (2.66 g, 16.39 mmol) in absolute EtOH (40 ml), was added with piperidine (2.43 ml, 24.58 mmol) portionwise, stirred at room temperature for 3h, cooled to  $0^\circ\text{C}$  and filtered. The precipitate was washed with EtOH, dried to yield product [6].

**(Z)-3-(4-Hydroxyphenyl)-2-(4-nitrophenyl)acrylonitrile (2):** Orange crystalline solid; Yield: 65%; m.p. 205–207  $^\circ\text{C}$ ; FT-IR/ATR: (KBr)  $\nu$  ( $\text{cm}^{-1}$ ): 3420, 2175, 1586, 1524, 1324;  $^1\text{H}$ NMR (400 MHz, DMSO- $d_6$ ,  $\delta/\text{ppm}$ ): 8.32 (d, 2H,  $J = 8.4$  Hz, Ar-H), 8.04 (s, 1H, -CH), 7.90 (d, 2H,  $J = 8.4$  Hz, Ar-H), 7.84 (d, 2H,  $J = 8.4$  Hz, Ar-H), 6.58 (d, 2H,  $J = 8.4$  Hz, Ar-H);  $^{13}\text{C}$  NMR (100 MHz, DMSO- $d_6$ ,  $\delta/\text{ppm}$ ): 161.8, 147.6, 145.4, 141.6, 133.2, 127.4, 124.6, 123.5, 117.6.

## 3 Results and discussion

### 3.1 Solvent effects on UV-Vis absorption maxima

As mentioned before,  $\pi$ -conjugated organic compounds have been broadly synthesized in recent years as an attractive platform for the design and manufacturing of a wide range of nano- and micro-structures for treatment in organic optoelectronics. Their relevant optical and electrical properties are considerably controlled by intermolecular interaction parameters associated with molecular packing and solute/solvent properties. Even though charge transfer is favored by dense ordered molecular stacking derived from strong  $\pi$ - $\pi$  interactions, corresponding intermolecular interactions often caused photoluminescence quenching due to concentration quenching phenomenon.

According to literature overview, the structural characteristics of cyanostilbens presented here are highly beneficial in providing corresponding electrical and photophysical properties on self-assembled materials. More precisely, these molecules possess a unique “elastic twist” character, regarding large torsional or conformational changes develop readily in response to light trigger and intermolecular interactions (Figure 2) [4,7].

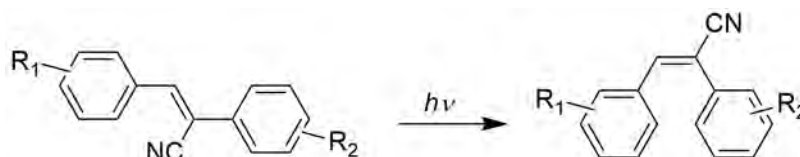


Figure 2. Chemical structures and isomeric change of  $\alpha$ -cyanostilbens

The UV-Vis absorption spectra of (Z)-2-(4-chlorophenyl)-3-(4-hydroxyphenyl)acrylonitrile (**1**) and (Z)-3-(4-hydroxyphenyl)-2-(4-nitrophenyl)acrylonitrile (**2**) were recorded in various polar protic and aprotic solvents and demonstrated in Figure 3 and Figure 4, respectively, while the corresponding absorption maxima were summarized in Table 1. The spectroscopic studies was performed at room temperature and the concentration of the solutions was  $1 \times 10^{-5}$  mol/dm $^3$ . Compound **1** possesses one absorption maxima in all polar protic solvents, while compound **2** possesses one absorption maxima in 2-butanol and 1-propanol and three absorption maxima in ethanol, which could be explained with better solute/solvent interactions, increased conjugation and intramolecular charge transfer. A bathochromic shift of compound **2** compared to compound **1** is also consequence of increased conjugation and intramolecular charge transfer inside this molecule. The absorption peak around 260–300 nm can be attributed to the  $\pi \rightarrow \pi$  transition in the aromatic part of the molecule [8,9].

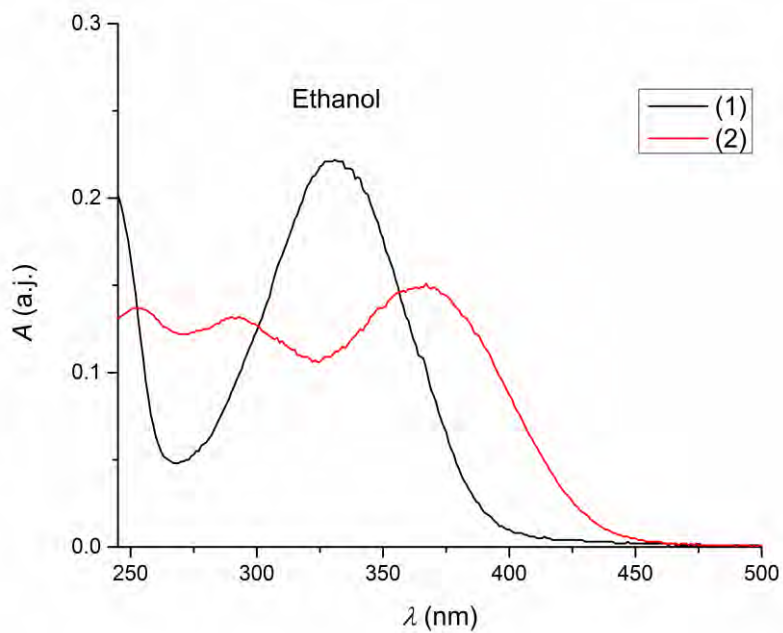


Figure 3. UV-Vis absorption spectra of the investigated compounds in polar protic solvent

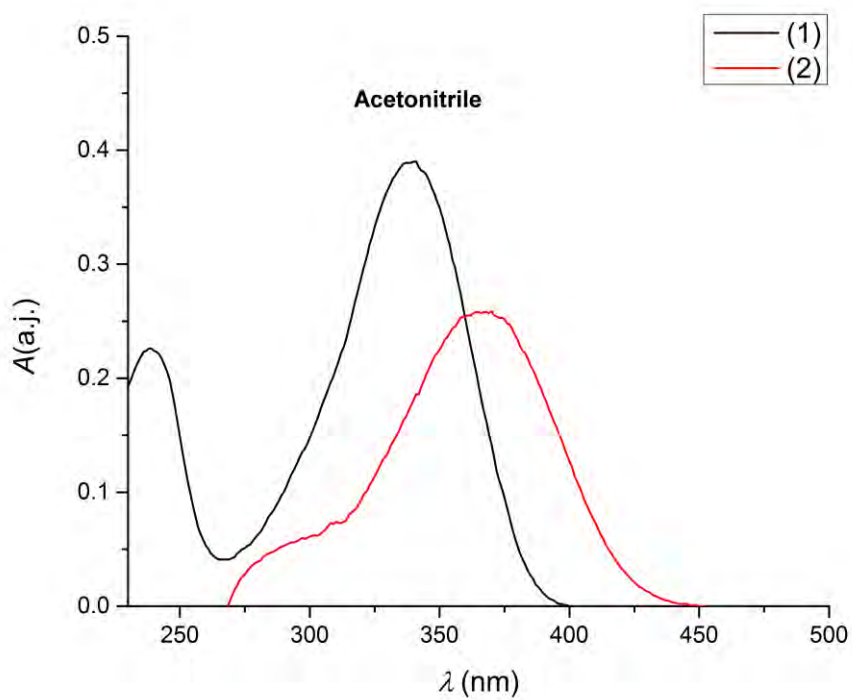


Figure 4. UV-Vis absorption spectra of the investigated compounds in polar aprotic solvent

Table 1. Absorption maxima of the investigated compounds in selected solvent set

<i>Solvent</i>	$\lambda$ (nm)-(1)	$\lambda$ (nm)-(2)
1. Acetonitrile	239, 339	238, 367
2. 2-Butanol	343	372
3. Chloroform	268, 335	251, 278, 361
4. Dichloromethane	237, 336	251, 358
5. Diethyl ether	237, 336	250, 358
6. Diisopropyl ether	345	395
7. 1,4-Dioxane	343	368
8. <i>N, N</i> -Dimethylformamide	343	384
9. Ethylacetate	340	290, 356
10. Ethanol	329	253, 292, 365
11. 1-Propanol	347	368
12. Methyl acetate	342	368
13. <i>N</i> -Methylformamide	342	310
14. <i>terc</i> -Butanol	347	368

To better understand nature of solute-solvent interactions, we applied the linear solvation energy relationship concept developed by Kamlet, Taft (equation 1) and Catalán (equation 2). These quantitative concepts separate the influence of non-specific chemical interactions, including electrostatic effects (dipolarity/polarizability), from specific interactions, i.e. hydrogen-bonding, which concerns to the molecular structure of a compound. The frequently employed simplified Kamlet–Taft equation applied to the UV/Vis absorption shift ( $\nu_{\max}$ ) of a solute is taking into account by equation (1), where  $\nu_{\max 0}$  is the solute property in a reference solvent, such as, a non-polar medium,  $\alpha$  describes the HBD (hydrogen-bond donating) ability,  $\beta$  the HBA (hydrogen-bond accepting) ability,  $\pi^*$  the dipolarity/polarizability of the solvents and  $a$ ,  $b$  and  $s$  are solvent-independent correlation coefficients that implies the contribution of various solvent effects to the UV-Vis absorption shift. To exceed imperfections of the Kamlet-Taft equation (dipolarity and polarizability of the solvent are included in one parameter), Catalán established three alternative empirical polarity scales: *SA*, *SB* and *SPP*, which are formally related to the Kamlet–Taft parameters  $\alpha$ ,  $\beta$  and  $\pi^*$ , respectively, while in 2004 Catalán and Hopf reported the fourth scale—the polarizability parameter *SP* measuring gradual differences in the surrounding’s polarizability. The preference of this extended concept is that each solvatochromic solvent parameter is established on a pair of well-defined reference homomorph solvatochromic probes. Another multiparameter equation, analogous to Equation (1), which involves two parameters for specific and two parameters for non-specific interactions (Equation (2)), has been pointed out that the *SPP* and *SP* scales are actually not independent of each other:

$$\nu_{\max} = \nu_{\max 0} + a\alpha + b\beta + s\pi^* \quad (1)$$

$$\nu_{\max} = \nu_{\max 0} + aSA + bSB + cSP + dSdP \quad (2)$$

To review this sensitivity and apart the individual solvation effects, the solvatochromic properties of these compounds were investigated in detail, and the coefficients of the individual interaction contributions were determined by using multiple correlation analysis [10].

Based on the results of multiple regressions presented in Tables 3 and 4, we can conclude that the absorption frequencies of the investigated  $\pi$ -conjugated organic compounds in the selected solvent set, demonstrated satisfactory correlation with  $\beta$ ,  $\alpha$ ,  $\pi^*$  parameters. The positive sign of the coefficient

$s$  in the total solvatochromic equations denotes a hypsochromic shift with enhancing solvent polarity/polarizability. In accordance with mentioned negative solvatochromism of the  $\pi$ -conjugated organic compounds are the positive signs of regression coefficient  $a$  and  $b$  denoting stabilization of the electronic excited state relative to ground state and *vice versa*. The percentage contributions of the solvatochromic parameters (Table 3) demonstrate that solvatochromism in general is the consequence of solvent basicity in the case of compound **1** and solvent dipolarity/polarizability in the case of the compound **2**. Based on the values of the correlation coefficient  $R$  and other statistical parameters, it can be concluded that the selected equations are suitable for the analysis of the solvatochromism of 4-substituted  $\alpha$ -cyanostilbens [11].

Table 2. Solvent parameters

Solvent	Kamlet-Taft			Catalán			
	$\pi^*$	$\beta$	$\alpha$	$SA$	$SB$	$SP$	$SdP$
1. Acetonitrile	0.75	0.31	0.19	0.044	0.286	0.645	0.974
2. 2-Butanol	0.40	0.80	0.69	0.221	0.888	0.656	0.706
3. Chloroform	0.58	0.00	0.44	0.047	0.071	0.783	0.614
4. Dichloromethane	0.82	0.00	0.30	0.040	0.178	0.761	0.769
5. Diethyl ether	0.24	0.47	0.00	0.000	0.562	0.617	0.385
6. Diisopropyl ether	0.27	0.49	0.00	0.000	0.657	0.625	0.324
7. 1,4-Dioxane	0.55	0.37	0.00	0.000	0.444	0.737	0.312
8. <i>N,N</i> -Dimethylformamide	0.88	0.69	0.00	0.031	0.613	0.759	0.977
9. Ethylacetate	0.55	0.45	0.00	0.000	0.542	0.656	0.603
10. Ethanol	0.54	0.77	0.83	0.400	0.658	0.633	0.783
11. 1-Propanol	0.52	0.90	0.78	0.367	0.782	0.658	0.748
12. Methyl acetate	0.60	0.42	0.00	0.000	0.527	0.645	0.637
13. <i>N</i> -Methylformamide	0.88	0.69	0.00	/	/	/	/
14. <i>tert</i> -Butanol	0.41	1.01	0.68	0.145	0.928	0.632	0.732

Table 3. Regression fits to solvatochromic parameters (**1**) and percentage contribution of solvatochromic parameters

No.	$\nu_0$ ( $10^3 \text{ cm}^{-1}$ )	$s$ ( $10^3 \text{ cm}^{-1}$ )	$b$ ( $10^3 \text{ cm}^{-1}$ )	$a$ ( $10^3 \text{ cm}^{-1}$ )	$R^a$	$s^b$	$F^c$	$P\pi$ (%)	$P\beta$ (%)	$P\alpha$ (%)
<b>1</b>	29.37 ( $\pm 0.17$ )	0.48 ( $\pm 0.21$ )	-0.91 ( $\pm 0.13$ )	0.24 ( $\pm 0.11$ )	0.941	0.129	21	29.45	55.83	14.72
<b>2</b>	21.92 ( $\pm 1.37$ )	8.28 ( $\pm 1.82$ )	2.52 ( $\pm 1.08$ )	-1.03 ( $\pm 0.24$ )	0.861	0.963	8	69.99	21.30	8.71

<sup>a</sup>Correlation coefficient; <sup>b</sup>Standard error of the estimate; <sup>c</sup>Fisher's test.

Table 4. Regression fits to solvatochromic parameters (2)

No.	$\nu_0$ ( $10^3\text{cm}^{-1}$ )	$a$ ( $10^3\text{cm}^{-1}$ )	$b$ ( $10^3\text{cm}^{-1}$ )	$c$ ( $10^3\text{cm}^{-1}$ )	$d$ ( $10^3\text{cm}^{-1}$ )	$R^a$	$s^b$	$F^c$
1	31.64 ( $\pm 1.26$ )	2.99 ( $\pm 0.77$ )	-1.71 ( $\pm 0.41$ )	-2.02 ( $\pm 0.35$ )	0.24 ( $\pm 0.04$ )	0.882	0.262	6
2	38.95 ( $\pm 2.05$ )	0.88 ( $\pm 0.17$ )	-1.83 ( $\pm 0.34$ )	-5.48 ( $\pm 0.42$ )	1.30 ( $\pm 0.43$ )	0.795	0.423	3

<sup>a</sup>Correlation coefficient; <sup>b</sup>Standard error of the estimate; <sup>c</sup>Fisher's test;

## 4 Conclusion

In this paper, two 4-substituted  $\alpha$ -cyanostilbene derivatives as potential molecular photoswitches, were synthesized and fully structurally characterized by determination of melting point, FT-IR,  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR, and UV-Vis spectra. The satisfactory correlations of the absorption frequencies with linear solvation models of Kamlet-Taft and Catalán demonstrate that the selected models give a correct interpretation of the linear solvation energy relationships of the complex  $\pi$ -conjugated system in the used solvent set. This also confirms that equation with three or four solvatochromic parameters can be used to evaluate the effects on both types of hydrogen bonding and the solvent dipolarity/polarizability effects on the relevant optical and electrical properties of  $\pi$ -conjugated organic compounds. For this reason, it is considered that the results presented in this work may be utilized to quantitatively separate the overall solvent effect into specific and nonspecific contributions using LSER method.

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