

Photoconductivity of Substituted Polyacetylenes†

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ABSTRACT

Photoconduction under visible light illumination is investigated using xerographic discharge technique in the photoreceptors of the following substituted polyacetylenes: $-[\text{HC}=\text{C}(\text{C}_6\text{H}_5\text{-}p\text{-R})]_n-$, $-[\text{HC}=\text{C}(\beta\text{-C}_4\text{H}_2\text{S-}\beta\text{-R}')]_n-$, and $-[\text{HC}=\text{C}[(\text{CH}_2)_m\text{-R}'']]_n-$, where $\text{R} = \text{CH}_3$ (**2**), $\text{CO}_2(\text{CH}_2)_6\text{OCO-Biph-OC}_7\text{H}_{15}$ (Biph = 4,4'-biphenyl; **3**); $\text{R}' = \text{Si}(\text{CH}_3)_3$ (**4**), Br (**5**); and $\text{R}'' = \text{CO}_2(\text{CH}_2)_6\text{OCO-Biph-OC}_9\text{H}_{19}$ ($m = 2$; **6**), 9-carbazolyl ($m = 3$; **7**), $\text{OCO-Biph-OC}_7\text{H}_{15}$ ($m = 9$; **8**). In the undoped state, 2–4 and 6–8 show much higher photosensitivity than poly(phenylacetylene) ($\text{R} = \text{H}$; **1**). The polyacetylenes with electron-donating and/or hole-transporting substituents perform better than do those with electron-accepting ones. The liquid crystalline polyacetylene **6** exhibits very high photosensitivity, probably because of the crystalline aggregates of its mesogenic pendants. C_{60} acts as a photoconductivity enhancer when doped to amorphous **3** but functions as a quencher when mixed with liquid crystalline **6**. While **3** shows low photosensitivity in the undoped state, doping with I_2 and sensitization with crystal violet dramatically

increase its photosensitivity up to $41.2 \times 10^{-3} \text{ lx}^{-1} \text{ sec}^{-1}$. Copyright © 2000 Society of Chemical Industry

KEYWORDS: substituted polyacetylenes; photoconductivity; liquid crystalline aggregates

INTRODUCTION

Xerography is a technology of choice for a wide diversity of photoelectronic applications. Of the many technologies that have led to the evolution of xerography, photoreceptor materials have played a major role [1–3]. Today, the development of new materials with novel photoconducting properties still holds the key to the further exploration of new technological applications of the photoimaging process.

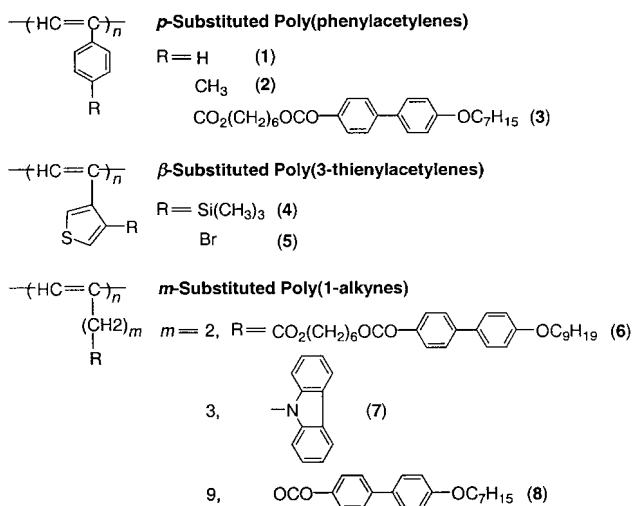
During the course of the exploration of molecular-complex photoreceptor systems, aggregate formation has been found to dramatically boost photosensitivity [1–3]. For example, when a homogeneous film of 4-(4'-dimethylaminophenyl)-2,6-diphenylthiapyrylium perchlorate, bis(4-diethylamino-2-methylphenyl)phenylmethane and polycarbonate is exposed to the vapor of dichloromethane, crystalline aggregates form, leading to a 100-fold increase in photosensitivity over its amorphous counterpart [4, 5]. The aggregate formation induced by the “wet” fuming process using the toxic halogenated solvent is, however, an obvious disadvantage of the thiapyrylium dye

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SCHEME 1.

photoreceptor system. It is well known that mesogenic groups in a thermotropic liquid crystal pack in an ordered fashion upon cooling from its molten state [6]. Thermal treatment of a liquid crystal with an appropriate transition temperature thus may be utilized as a simple "dry" process to induce the formation of crystalline aggregates and hence to enhance the efficiency of photoconduction. Such a possibility, however, has not been explored.

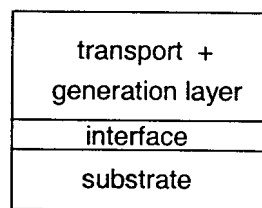
Conjugated polymers have attracted much interest among scientists and technologists because of their intriguing potential applications in optics and electronics systems. Polyacetylene is a prototypical conjugated polymer and shows high photoconductivity [7]. Unfortunately, however, polyacetylene is intractable and unstable, which makes it difficult to find practical applications. The extensive synthetic efforts in the past two decades have created a variety of processible and stable polyacetylene derivatives by attaching substituent pendants to the polyacetylene backbone ("substituted polyacetylenes") [8, 9], some of which have been found to possess unique materials properties such as γ -radiolysis susceptibility [10–12], optical activity [13], gas permeability [14, 15] and pervaporation capacity [16, 17]. In our previous studies, we investigated liquid crystallinity [18–21], light emissivity [22–25], chirality/helicity [24, 26, 27], optical nonlinearity [24, 28] and self-organization properties [24, 26, 29] of the substituted polyacetylenes recently synthesized in our laboratories. In this work, we studied photoconduction in the polyacetylenes in their undoped and doped states (Scheme 1).

EXPERIMENTAL

Materials and Instrumentation

All the polyacetylenes used in this study, that is, the *p*-substituted poly(phenylacetylenes) **1–3**, the *β*-substituted poly(3-thienylacetylenes) **4–5**, and the

Single-Layer Photoreceptor



SCHEME 2.

m-substituted poly(1-alkynes) **6–8**, were synthesized according to our previously published procedures [20, 30, 31]. Poly(9-vinylcarbazole) (PVK) was prepared in our laboratories following a published polymerization procedure [32]. 4-(Diethylamino)benzaldehyde 1-(1'-naphthyl)-1-phenylhydrazine (DENPH), polycarbonate (PC), polyamide (PAM), C₆₀, I₂ and crystal violet (CV) were all purchased from commercial sources. All the organic solvents used in this study were of spectroscopic grades and purified by distillation prior to use.

Differential scanning calorimetry (DSC) thermograms were recorded on a Setaram DSC 92 calorimeter under nitrogen at a heating or cooling rate of 10°C/min. An Olympus BX 60 polarized optical microscope (POM) with a Linkam TMS 92 hot stage was used in cross-polarized mode for the visual observation of mesophasic textures of the liquid crystalline polymers. X-ray diffraction (XRD) patterns were recorded on a Philips PW 1830 powder diffractometer using the monochromatized X-ray beam from the nickel-filtered Cu *K* α radiation with a wavelength of 1.5406 Å. UV spectra were recorded on a Milton Roy Spectronic 3000 Array spectrophotometer in tetrahydrofuran (THF) and fluorescence spectra were measured in chloroform on an SLM Aminco JD-490 spectrofluorometer. Film thickness was evaluated using an Elektro-Physik Minitest 2000 thin film thickness measurement system. Photosensitivity of the photoreceptor systems was measured on a GDT-II photoconductivity measurement device using a 5W, 24V incandescent lamp as light source, whose emission is mainly in the visible spectral region (~400–800nm) [33]. The light with monochromatic wavelength of 500, 573, 601 or 762nm was isolated from the lamp using optical filters.

Photoreceptor Preparation

A single-layer photoreceptor based on an undoped polyacetylene was prepared by coating a 1,2-dichloroethane solution of a substituted polyacetylene, DENPH, and PC (3:40:40 by weight) onto an electrically grounded aluminum substrate that had been precoated with a thin interface layer of PAM (Scheme 2). The thickness of the photoreceptor film was 20–30 μ m. The photoreceptor was heated at 80°C in an oven for several hours to remove the residual solvent. In the photoreceptor system, the

TABLE 1. Photoconductivity of Substituted Polyacetylenes^a

No.	Polymer	V_o (V)	R_d (V/sec)	ΔV_1 (%)	$t_{1/2}$ (sec)	V_r (V)	S (10^{-3} $1x^{-1} sec^{-1}$)
<i>Poly(phenylacetylenes)</i>							
1	1	781	10	13	6.313	292	2.64
2	2	816	12	16	4.438	203	3.76
3	3	808	10	14	5.813	282	2.80
<i>Poly(3-thienylacetylenes)</i>							
4	4	736	19	28	2.375	154	6.99
5	5	707	13	7	>10	339	<1.67
<i>Poly(1-alkynes)</i>							
6	6	707	20	40	1.438	98	11.63
7	7	696	18	35	1.750	106	9.52
8	8	656	18	22	3.063	117	5.43
<i>Poly(9-vinylcarbazole) (PVK)^b</i>							
9	PVK	700	9	8	8.625	342	1.93

^a Photoreceptors of single-layer configuration were exposed to a 573 nm light with an intensity of 60 lx.

^b For the purpose of comparison.

V_o = initial surface charge potential, R_d = dark decay, ΔV_1 = percentage of potential discharge after 1 sec of exposure, $t_{1/2}$ = half-discharge time, V_r = residual surface charge potential, S = photosensitivity.

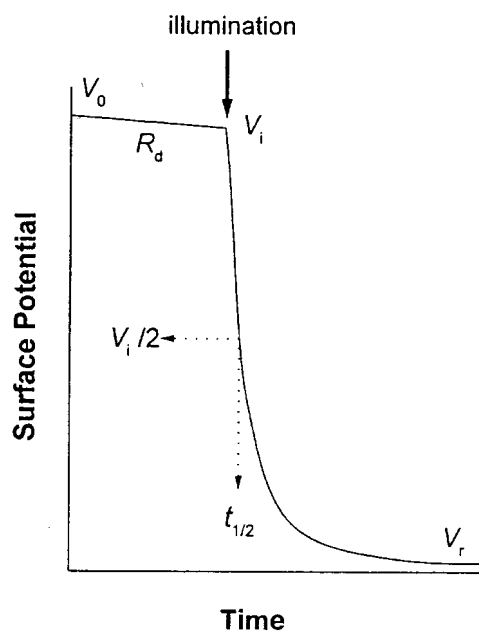


FIGURE 1. A typical photoinduced xerographic discharge curve, where V_o is initial surface charge potential, R_d is dark decay, V_i is surface potential at illumination, $t_{1/2}$ is half-discharge time and V_r is residual surface charge potential.

polyacetylene functions as a charge generation material and DENPH serves as a charge transport material (while PC is the most widely used polymer matrix in photoreceptor systems). A

single-layer photoreceptor consisting of a doped or sensitized substituted polyacetylene was prepared in the similar fashion with the addition of a dopant or sensitizer.

Photoconductivity Measurement

Photoconductivity of the polyacetylene photoreceptors was evaluated using the standard photo-induced discharge technique, which is the basis of the xerographic process in the photocopying systems [34]. The photoreceptor was first negatively corona-charged to a surface potential V_o and then kept in the dark for 3 sec. Upon exposure to a light of intensity I , the electron-hole pairs form. A fraction of the pairs separate and migrate to the free surface and substrate electrode. The surface charge is thus dissipated by neutralization. From the photoinduced discharge curve illustrated in Fig. 1, the following photoconduction parameters can be obtained: dark decay ($R_d = (V_o - V_i)/3$, where V_i is the surface potential right before illumination), percentage of potential discharge in 1 sec ($\Delta V_1 = [(V_i - V_1)/V_i] \times 100\%$, where V_1 is the surface potential after 1 sec exposure), time from V_i to its half value under exposure ($t_{1/2}$), residual surface charge potential (V_r) and half-discharge exposure energy ($E_{1/2} = t_{1/2} \times I$). Photosensitivity (S) is defined as the reciprocal of the half-discharge exposure energy ($S = 1/E_{1/2}$).

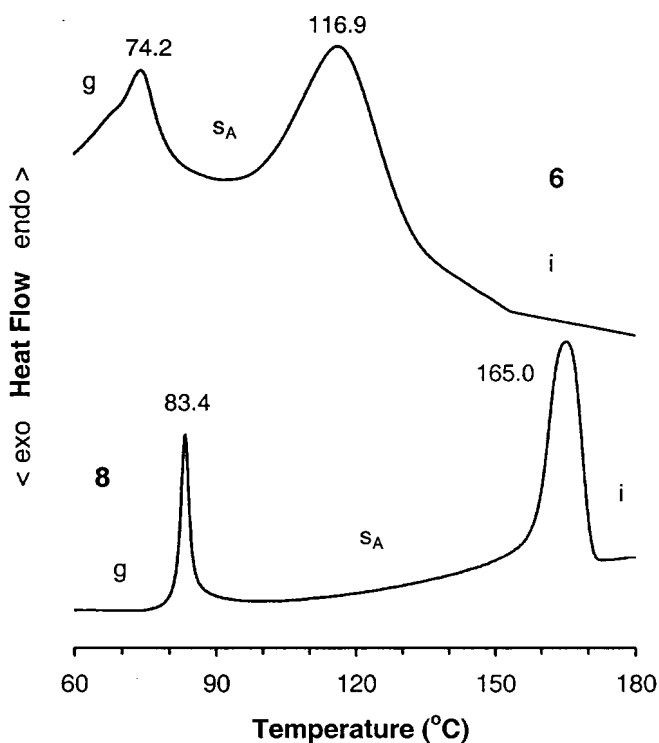


FIGURE 2. Thermal transitions of liquid crystalline polyacetylenes **6** and **8**. While the DSC thermograms were taken for both the 1st cooling and 2nd heating scans, only the data for the later are shown here. The phase transitions were confirmed by POM and XRD analyses.

RESULTS AND DISCUSSION

Photoconductivity of Pure (Undoped) Polyacetylenes

The photoconductivity of single-layer photoreceptors of the undoped polyacetylenes along with that of PVK exposed to a visible light of 573 nm is summarized in Table 1. The photoreceptor of PPA (**1**) can readily be negatively charged to a surface potential of 781 V, which slightly decreases to 751 V in 3 sec (V_i) in the dark, giving a low dark decay (R_d) of 10 V/sec (Table 1, no. 1). After exposure to the visible light of 60 lx for 1 sec, V_i decreases to 653 V, achieving a 13% potential discharge (ΔV_1). The surface potential drops to its half value in ~ 6 sec ($t_{1/2}$). The residual charge potential (V_r) left on the photoreceptor surface is 292 V and the photosensitivity (S) of **1** is estimated to be $2.64 \times 10^{-3} \text{ lx}^{-1} \text{ sec}^{-1}$. When an electron-donating group of methyl is introduced to the *para* position of the phenyl ring (**2**), the R_d and ΔV_1 values change little. The $t_{1/2}$ and V_r values, however, decrease and, as a result, S value increases. When an electron-accepting group of ester is introduced (**3**), the photosensitivity of the photoreceptor slightly increases. This is different from the previous observation by other researchers (incorporation of an acceptor group leading to a decrease in photosensitivity [35]). It is known that **3** is liquid crystalline and its melting transition temperature is

quite high (135.5°C) [20]. The high rigidity of the PPA backbone distorts the packing arrangements of the liquid crystalline mesogens, but there might still be a small portion of the mesogenic groups that have aggregated. The competition between the constructive (aggregation) and destructive (electron withdrawing) effects may give rise to the observed slight increase in the photosensitivity.

The poly(3-thienylacetylene) with a donor group of trimethylsilyl at the β position (**4**) shows a fast ΔV_1 (28%), a short $t_{1/2}$ (~ 2 sec), a low V_r (154 V), and a high S value ($\sim 7 \times 10^{-3} \text{ lx}^{-1} \text{ sec}^{-1}$; Table 1, no. 4). In contrast, the polymer with an acceptor group of bromo (**5**) performs poorly as a photoreceptor with its $t_{1/2} > 10$ sec, $V_r > V_i/2$, and $S < 1.67 \times 10^{-3} \text{ lx}^{-1} \text{ sec}^{-1}$. It is known that polysilanes are hole transporting [1]. The trimethylsilyl moiety attached to the thienyl ring in **4** may take part in the charge carrier transport processes, facilitating the movement of the holes toward the surface of the photoreceptor for neutralization. Combination of the electron-donating and hole-transporting effects thus makes **4** an excellent photoconductor.

Compared with the above-discussed two groups of polyacetylenes, the poly(1-alkynes) **6–8** are generally better photoreceptors. The high photosensitivity of **7** may be due to the synergetic contribution of the electron-donating propyl spacer and the hole-transporting carbazolyl group. The pendant group of **6** is structurally similar to that of **8**, but the photosensitivity of the former is more than 2-fold higher than that of the latter. While both **6** and **8** are liquid crystalline, their melting transition temperatures are different: 74.2°C for **6** and 83.4°C for **8** (Fig. 2). When **6** is heated at 80°C in the photoreceptor preparation process, the fluidity in the molten sample of **6** allows the mesogenic groups to move together to self-align in an ordered s_A phase. Cooling the liquid crystalline **6** to room temperature further enhances the ordering of the mesogenic pendants, and the crystalline aggregates will boost the photosensitivity of the polymer. On the other hand, at 80°C, **8** is still in the glassy state. There might be some ordering, but its extent should be quite small. Thus unlike its liquid crystalline cousin **6** with a lower melting transition temperature, there is no marked aggregation effect observed in the photoreceptor of **8**.

Compared to PVK, **6** displays a 6-fold higher photosensitivity in the undoped state, thanks to its conjugated backbone consisting of alternating double bonds. It is generally agreed that for xerographic applications, a photoreceptor should meet the following criteria: (1) chargeable to a high surface potential and having a low rate of thermal generation of free carriers, (2) possessing a high sensitivity in the visible and/or near infrared spectral region, and (3) dissipating the surface charge at a fast rate to a high extent upon exposure to light. The photoreceptor of **6** meets all of these requirements; it must be stressed that all of these remarkable characteristics are for the pure polymer in its undoped state. Thus **6** is an excellent

TABLE 2. Photoconductivity of C₆₀-Doped Polyacetylenes (PAs)^a

No.	PA	C ₆₀ (wt %)	Wavelength (nm)	<i>l</i> (1x)	V _o (V)	R _d (V/sec)	ΔV ₁ (%)	t _{1/2} (sec)	V _r (V)	S (10 ⁻³ lx ⁻¹ sec ⁻¹)
1	3	0	visible	800	882	18	48	1.063	140	1.18
2		1.5	visible	800	621	33	77	0.313	82	4.00
3			500	60	562	15	27	2.625	109	6.35
4			573	60	636	18	36	1.750	136	9.52
5			601	60	585	10	18	4.313	148	3.86
6			762	60	554	11	15	5.188	171	3.21
7	6	0	visible	800	750	46	70	0.438	113	2.86
8		1.5	visible	800	492	13	46	1.190	117	1.05
9			500	60	445	6	10	8.000	183	2.08
10			573	60	503	6	10	7.625	203	2.19
11			601	60	503	5	6	>10	285	<1.67
12			762	60	498	5	3	>10	358	<1.67
13	8	0	visible	800	623	62	52	0.875	131	1.43
14		1.5	visible	800	539	34	75	0.375	70	3.33
15			500	60	527	16	22	3.125	97	5.33
16			573	60	589	15	25	2.750	101	6.06
17			601	60	609	15	18	4.063	128	4.10
18			762	60	664	14	7	>10	300	<1.67

^a Single-layer photoreceptors were exposed to an incandescent lamp, whose emission is mainly in the visible spectral region (~400–800 nm), or to a monochromatic light with wavelength of 500, 573, 601 or 762 nm, which was isolated from the lamp using optical filters. *l*: exposure intensity. Other abbreviations as in Table 1.

photoreceptor material with great commercial potential.

The polyacetylenes in Table 1 can be divided into two categories: one with donor groups (**2**, **4**, **6**, **7** and **8**) and another one with acceptor groups (**1**, **3** and **5**). Obviously, the former shows higher photosensitivity than the latter. For the liquid crystalline polymers (**3**, **6** and **8**), the photosensitivity changes according to whether the morphologic and electronic effects are working in a synergistic or antagonistic fashion. When the morphologic effect is opposite to the electronic one, the photosensitivity remains to be low (**3**). When the two effects are both constructive, it leads to large enhancements in the photosensitivity (**6** and **8**).

Photoconductivity of C₆₀-doped Polyacetylenes

It was found that C₆₀ doping of PVK [34] and poly(*p*-phenylenevinylene) derivatives [36] enhanced photoconductivity of the polymers because of the photoinduced electron transfer from the polymers to C₆₀.

Our results show that doping of a small amount of C₆₀ (1.5 wt%) into **3** decreases the half-discharge time (*t*_{1/2}) and the residual potential (*V*_r; Table 2). In fact, the C₆₀ doping helps dissipate the charge potential at a fast rate to a lower level. So the *S* value increases by a factor of ~4. The photoreceptor shows quite high photosensitivity in all the wavelengths investigated, with the highest *S* value (9.52 × 10⁻³ lx⁻¹ sec⁻¹) obtained at 573 nm.

When **6** is doped with C₆₀, however, its

photosensitivity drops to about one-third of that of the undoped one in the visible spectral region (Table 2, nos 7 and 8). The photoconductivity at 601 or 762 nm is so low that its *S* value cannot be accurately determined. Interestingly, however, when **8** is doped with C₆₀, an increase in the photosensitivity is observed, although the extent of the increase is not as large as that observed as in doped **3**.

UV absorption and fluorescence emission spectra of the undoped and doped polymers are measured. Not much change, however, is observed in both the absorption and emission spectra before and after doping, implying that the charge transfer complexes between the polymers and C₆₀ may have not formed in the ground states. All of the three polyacetylenes (**3**, **6** and **8**) are liquid crystalline but with different melting transition temperatures (135.5, 74.2 and 83.4 °C, respectively). Because of the distorting effect of the rigid PPA backbone, the mesogenic groups in **3** cannot pack well and thus the polymer predominantly adopts an amorphous state [20]. The melting transition temperature of **3** is very high, and the heating at 80 °C cannot bring the mesogenic groups into liquid crystalline state. The polymer/fullerene heterojunction interfaces thus effectively enhances the photoconductivity of the photoreceptor through electron transfer and charge separation processes [34, 36].

It was reported by Edman and co-workers that addition of a very small amount of C₆₀ to a mixture of poly(ethylene oxide) and LiCF₃SO₃ readily breaks up the crystalline structure of the polymer-salt complexes [37]. Our previous work

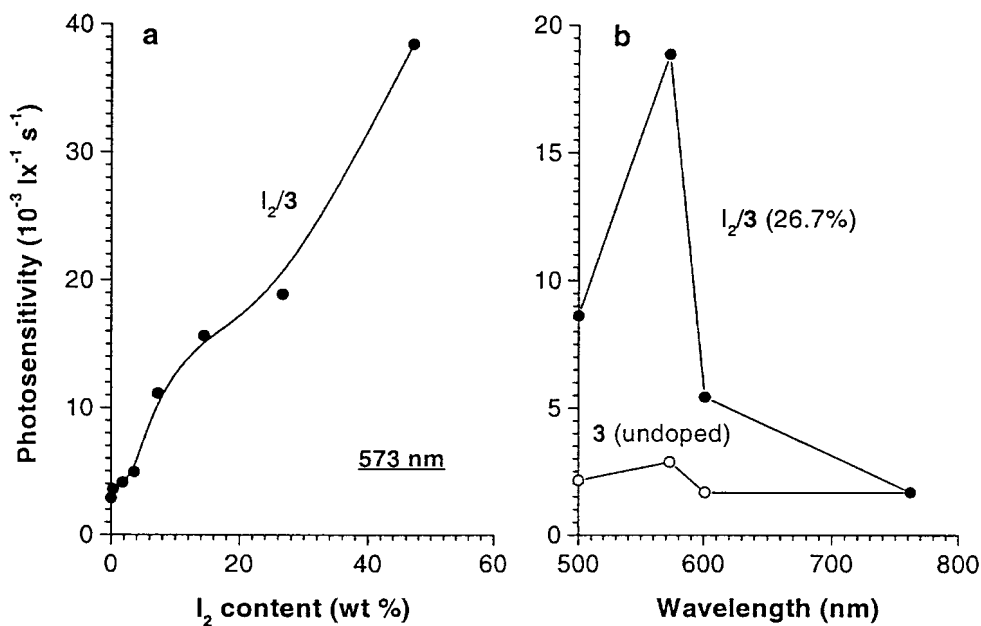


FIGURE 3. Effects of (a) iodine content and (b) illumination wavelength on the photosensitivity of single-layer photoreceptors of **3** exposed to visible light of 60 lx.

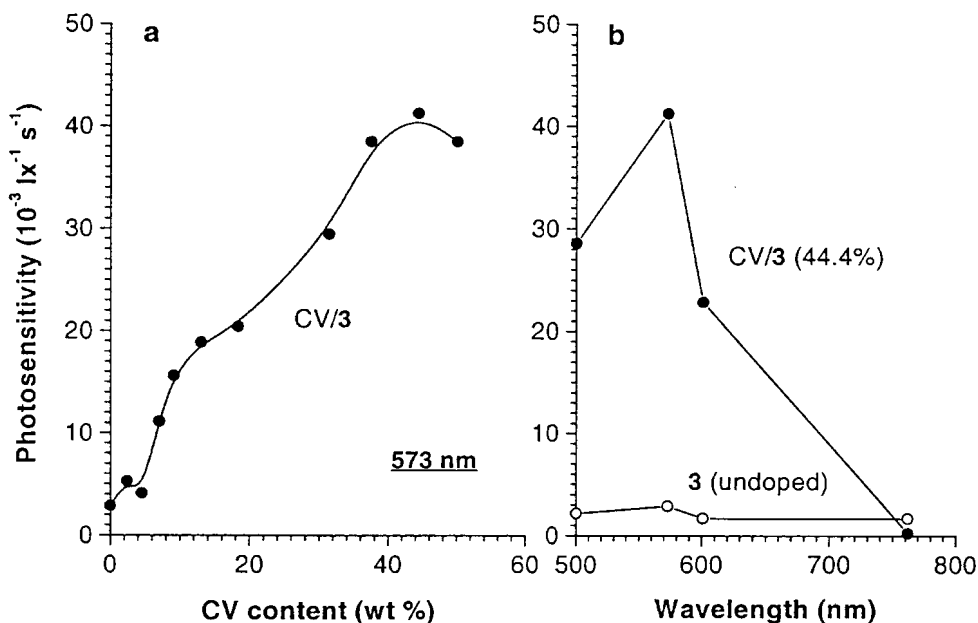


FIGURE 4. Effects of (a) crystal violet content and (b) illumination wavelength on the photosensitivity of single-layer photoreceptors of **3** exposed to visible light of 60 lx.

[38,39] has revealed that the giant three-dimensional C_{60} cages act as plasticizers in the PC systems, which increases the free volumes between the polymer chains, enhances the local segmental flexibility, and consequently lowers the glass transition temperature of the C_{60} -containing polymers. In the case of **6**, whose melting transition temperature is below 80°C , the molten polymer "liquid" may dissolve the C_{60} molecules. Upon cooling, the molecularly dispersed bulky C_{60} cages may act as "impurities" to hamper the packing of

the liquid crystal mesogens, leading to a large decrease in the photosensitivity.

The temperature of melting transition of **8** is close to that employed in the drying process of the photoreceptor films. Thus the two antagonistic effects compete in the photoreceptor system: part of the buckyballs may act as the crystallinity-breaking plasticizers while another part of the C_{60} molecules may aggregate in the amorphous region of the polymer. The result of the competition is the slight increase in the photosensitivity.

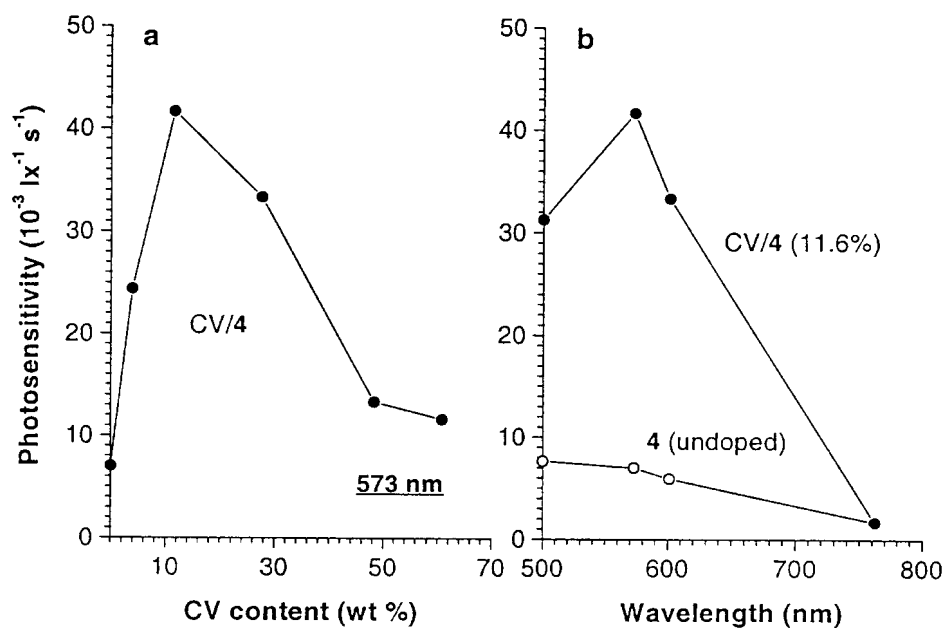


FIGURE 5. Effects of (a) crystal violet content and (b) illumination wavelength on the photosensitivity of single-layer photoreceptors of **4** exposed to visible light of 60 lx.

Photoconductivity of I₂-doped and CV-sensitized Polyacetylenes

In order to boost the relatively low photoconductivity of **3** in the pure state, I₂ was used as a dopant. As shown in Fig. 3(a) the photosensitivity of the photoreceptors of I₂-doped **3** to the 573 nm illumination monotonically increases with the increase in the I₂ content. The increase in the high I₂ content region becomes sharper, possibly because of the more efficient iodine aggregation leading to the formation of heterojunctions with larger interface areas. At ~46% I₂ doping, the photosensitivity is as high as $\sim 38 \times 10^{-3} \text{ lx}^{-1} \text{ sec}^{-1}$, which is ~14 times higher than that of the undoped form. The shapes of the photosensitivity spectra of the photoreceptors of the undoped and doped **3** (with 26.7% I₂) are similar, both giving a maximum at 573 nm (Fig. 3b). The magnitudes of the photosensitivity of the doped **3** are, however, much higher in most part of the visible spectral region.

The photoconductivity of CV-sensitized **3** at 573 nm is depicted in Fig. 4. The enhancement in the photosensitivity of the sensitized **3** may involve efficient electron transfer from the donor molecule (CV) to the photoexcited **3** [40]. The photosensitivity spectra of the sensitized and unsensitized **3** again have the similar shapes, with the former locating well above the latter in most of the visible region. At 573 nm, the sensitized **3** shows a photosensitivity ~15 times higher than that of the unsensitized one.

Similar results are obtained when **4** is sensitized by CV (Fig. 5). However, the photosensitivity of the CV-sensitized **4** at 573 nm reaches its maximum at a much lower CV content (~12%; Fig. 5a). The CV-sensitized **4** with this relatively small amount of sensitizer exhibits quite high photoconductivity in

the spectral region around 700 nm (Fig. 5b). For a material to be used in digital xerography, its photosensitivity in the red and infrared regions is usually required [1–3]. The CV-sensitized **4** with the infrared sensitivity thus may find potential applications in the digital photoimaging technologies.

CONCLUSION

The following structure–property relationships have been observed in the polyacetylene photoreceptor systems: (i) the polymers with donor substituents (**2**, **4**, **6–8**) show higher photosensitivity than those bearing acceptor ones (**3**, **5**); (ii) when the donor substituents are simultaneously hole transporting (**4**, **7**), the photosensitivity further increases; and (iii) when the donor substituent is mesogenic and can be packed in an ordered fashion (**6**), the photoconduction becomes very efficient.

A simple thermal treatment on the liquid crystalline polyacetylene with an appropriate transition temperature (**6**) has been found to boost its photosensitivity. The “dry” process for inducing the formation of crystalline aggregates is an new approach, with an obvious advantage of obviating the use of the toxic solvent in the “wet” fuming process in the preparation of conventional photoreceptors of dye aggregates [1–3]. Polymer **6** possesses both liquid crystalline and photoconductive properties and may find applications in spatial light modulating systems [40].

C₆₀ has been so far reported to function as a very efficient photoconductivity enhancer [34, 36]. We have, however, found in this study that C₆₀ is not a universal enhancer: it acts as either a photoconductivity promoter or an aggregation-breaking

plasticizer, depending on the morphology of the conjugated polymers.

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