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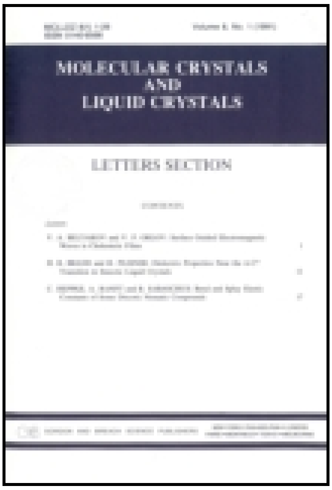

Molecular Crystals and Liquid Crystals
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl20

Photoelectric Properties of Photoconducting Composites Based on Non-Photoconducting Polymers Doped by Triarylmethane and Xanthene Dyes

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Published online: 28 Mar 2014.

To cite this article: S. L. Studzinsky (2014) Photoelectric Properties of Photoconducting Composites Based on Non-Photoconducting Polymers Doped by Triarylmethane and Xanthene Dyes, Molecular Crystals and Liquid Crystals, 589:1, 183-194, DOI: 10.1080/15421406.2013.872822

To link to this article: http://dx.doi.org/10.1080/15421406.2013.872822

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Mol. Cryst. Liq. Cryst., Vol. 589: pp. 183–194, 2014
Copyright © Taylor & Francis Group, LLC
ISSN: 1542-1406 print/1563-5287 online
DOI: 10.1080/15421406.2013.872822

Photoelectric Properties of Photoconducting Composites Based on Non-Photoconducting Polymers Doped by Triarylmethane and Xanthene Dyes

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The films of polymeric composites based on non-photoconducting polyvinyl alcohol and polystyrene doped by various concentration of xanthene and triarylmethane type dyes have been prepared. Their photophysical, electrical, and spectral properties have been investigated. It was shown, that all studied composite films exhibit photovoltaic properties and anomalous negative photoconductivity effect. The diffusion nature of observed photovoltaic effect was established. The features of photovoltaic effect and photoconductivity, as well as the possible negative photoconductivity mechanism are discussed.

Keywords Polymeric composite films; xanthene and triarylmethane dyes; photovoltaic effect; negative photoconductivity

Introduction

Polymer composite films (PCFs) with added organic dyes are commonly used in digital recording and electroluminescence media as well as in photoelectric solar energy converters [1–9]. Monomeric dye molecules in PCF act as sites for light absorption and the photogeneration of nonequilibrium charge carriers. Considerable progress has been achieved in controlling the photophysical and electrophysical properties of PCF by varying the chemical structure of the dye and polymer. However, these properties also depend on the geometric dimensions of the PCF components (geometry and size of their possible associate species) [5–10]. Besides, such dependence is often related to the transformation of the energy structure of the dopants in a polymer binder. Thus, there is theoretical and practical interest in creating new photoactive media derived from organic dyes doped colored PCFs. Xanthene and triarylmethane dyes hold the greatest interest among organic dyes as the light energy converters and as the photoconductive materials components since these dyes have the high extinction coefficients and absorb light in a broad spectral range [5, 8, 9, 11]. In the present work, we studied the photoconductivity, photovoltaic and photodielectric properties of the non-photoconducting polymers based PCFs doped with various concentration of an ionic dyes of a different types – Rhodamine B and Gentian violet.

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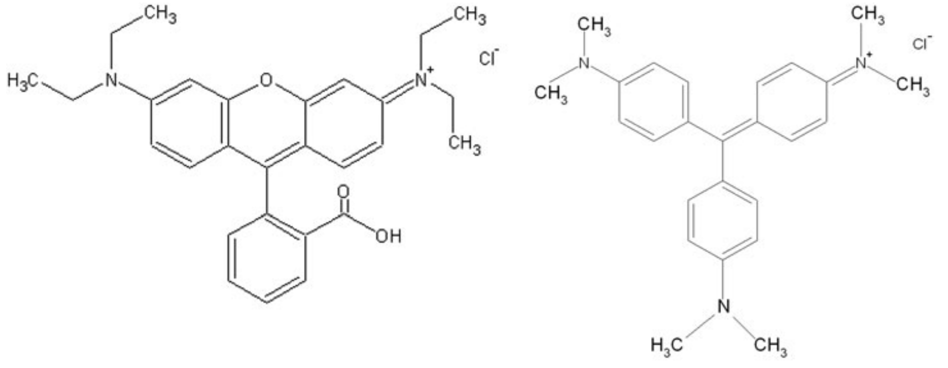
Samples and Experimental Methods

Non-photoconducting polystyrene (PS) and polyvinyl alcohol (PVA) were used as a polymer-matrixes and the xanthene type ionic dye Rhodamine B (Rd), as well as triarylmethane type ionic dye Gentian violet (Gn) were used as a photosensitive components of the PCFs studied (Scheme 1).



Rhodamine B (Rd) Gentian violet (Gn)

Scheme 1. Molecular structures of studied dyes.

The samples were prepared as structures with a free surface [glass substrate – PCF], [glass substrate – transparent electroconducting ITO (SnO_2:In_2O_3) layer – PCF], and as the sandwich structures. The sandwich structures were prepared both as the glass substrate – ITO layer – PCF – Ag-electrode samples and as the symmetrical type [glass – ITO – PCF – ITO - glass] sandwich structures. Concentrations of dyes in all films were 1 or 10 wt%. The polymeric composite films were formed by pouring-evaporating of dye-polymer solutions (water solutions in the PVA case and dichloromethane solutions in the PS case, respectively) at the substrates. The PCF films thickness was 1.0–2.0 μm.

The techniques for measuring optical density (D) (in the samples with free surface) in the wavelength range 400–900 nm, dark (j_d) and photocurrent density (j) were described in our previous work [2, 12, 13]. An incandescent lamp with glass light filters was used as a light source to study the photoconduction properties of the PCFs. The light intensity (I) was varied by neutral light filters in the range 1–50 W/m^2. The samples with the sandwiched structure (ITO-PCF-Ag type) were used for measurements of the conductivity current density before light irradiation (j_d) and the current density in the course of irradiation (j) as a function of the electric voltage (U) applied to the electric contacts, irradiation time (t), and after switching off light. The j_d value was determined after switching on an external electric voltage and establishing transient processes. The j value was determined in the course of light irradiation, and the j/j_d value was calculated. The irradiation was performed at the side of the conducting ITO layer. The electric voltage (U) applied to the electrical contacts in the sandwich-structure samples was varied in the range 1–120 V.

Photovoltage (surface) was measured by the modified Kelvin method [14] in the samples with a free surface (under illumination of the transparent ITO-electrode side). The additional photovoltaic measurements for the investigated PCFs were carried out in the symmetrical type (glass – ITO – PCF – ITO – glass) sandwich structure samples by used high resistance voltmeter based on CA3130 voltage follower scheme [l4]. The kinetics of photocurrent j and photovoltaic response (V_{PH}) were measured using a storage oscilloscope Tektronix TDS1001B and a storage USB-oscilloscope BM 8020.

The samples with the sandwiched structure (ITO-PCF-Ag type) were also characterized by the dielectric loss tangent (tanδ) and electric capacitance (C), which were measured in the dark and on exposure using a sinusoidal alternating voltage with an effective magnitude of 6 V and frequencies within $f = 10^3$–10^4 Hz. The results of these measurements were used to calculate the values of $\Delta\tan\delta_{PH} = (\tan\delta_{PH} - \tan\delta_0)/\tan\delta_0$ and $\Delta C_{PH} = (C_{PH} - C_0)/C_0$, where $\tan\delta_{PH}$, C_{PH}, and $\tan\delta_0$, C_0 are the loss tangent and capacitance after and before switching on the light. The photodielectrical measurements were carried out using a dielectric coefficient and loss factor testing set of the TR-9701 type (based on Schering bridge scheme), external sinus signal generator G3–33 as the alternating voltage source and F-582 null-indicator according to [14–16]. As a photoexcitation light source for the photovoltaic and photodielectric measurements, we used a white (3 mW) semiconductor light-emitting diode (508H245WC-2.2-MD). All measurements were carried out at room temperature.

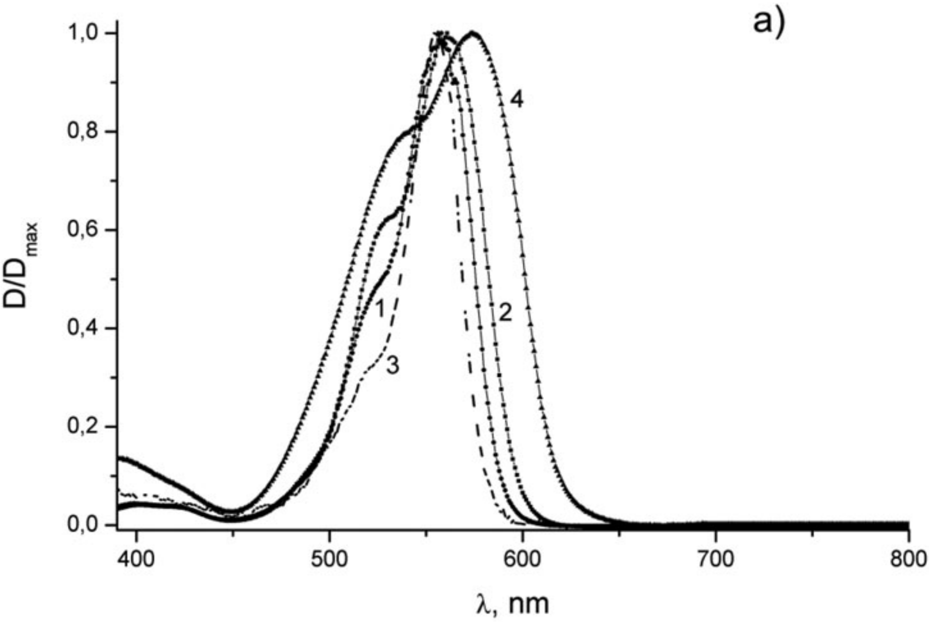
Results and Discussion

The film polymeric composites based on non-photoconducting polyvinyl alcohol and polystyrene doped by various concentration both of Rhodamine B and Gentian violet dyes have been obtained. The films of pure PVA and PS are transparent in the visible and near infrared regions, and they haven't photoconductivity, photovoltaic and photodielectric properties in this area. In the polymeric films which contain dye additives, the bands of absorption (Figure 1a, b) are defined by excitation and relaxation of the excited states of dye's molecules [9, 11].

Figure 1 (a, b) shows that under increasing the dyes concentration in the PCFs, the absorption spectra showed strong broadening of the absorption bands in comparison with the corresponding absorption spectra of low dye concentration doped PCFs. The nature of the spectral changes depends on the dye concentration. In a PCFs the absorption intensity in the short-wavelength spectral region increases with dye concentration in the PCFs increasing. We may also conclude that an increase in the concentration of dyes in PCF causes association of its molecules [9, 11]. The concentration dependent PCFs absorption spectra change indicates that the absorption band is nonuniformly broadened due to the superposition of bands of various dye forms [11]. Both the Rd and Gn monomer and its aggregates are most likely these forms in concentrated polymer solutions [11].

It was found, that all studied composite films exhibit photovoltaic effect and photoconductivity under the photoexcitation in the dye absorption region. The kinetics of the surface photovoltage growth and relaxation in all prepared PCFs samples with a free surface are presented in Figure 2. The maximal absolute value of photovoltage (∼ 035–0,5 V) is approximately the same as that for all investigated PCFs (Figure 2c), but the kinetics and characteristic time constants of photovoltaic effect growth and dark relaxation are different for a series of investigated PCF-samples (Figure 2a, b).

Figure 2 demonstrates the main features of photovoltaic response kinetics in the investigated PCFs. The growth and relaxation of photovoltaic responses V_{PH} upon the light being switched on and off, respectively (Figure 2), is characterized by slow kinetics with a relaxation time constant, which is more than ten times greater than the time constant of V_{PH} growth during the PCFs photoexposition.



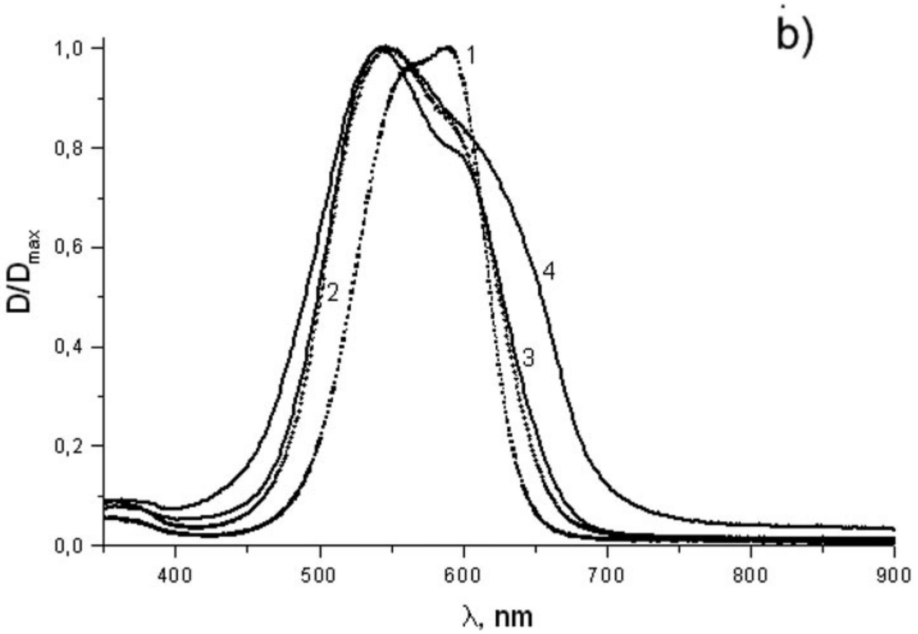


Figure 1. a) Normalized spectra for absorption of PVA (1, 2) and PS (3, 4) based PCF with added 1 wt.% Rd (1, 3), and 10 wt.% Rd (2, 4); b) normalized spectra for absorption of PVA (1, 2) and PS (3, 4) based PCF with added 1 wt.% Gn (1, 3), and 10 wt.% Gn (2, 4).

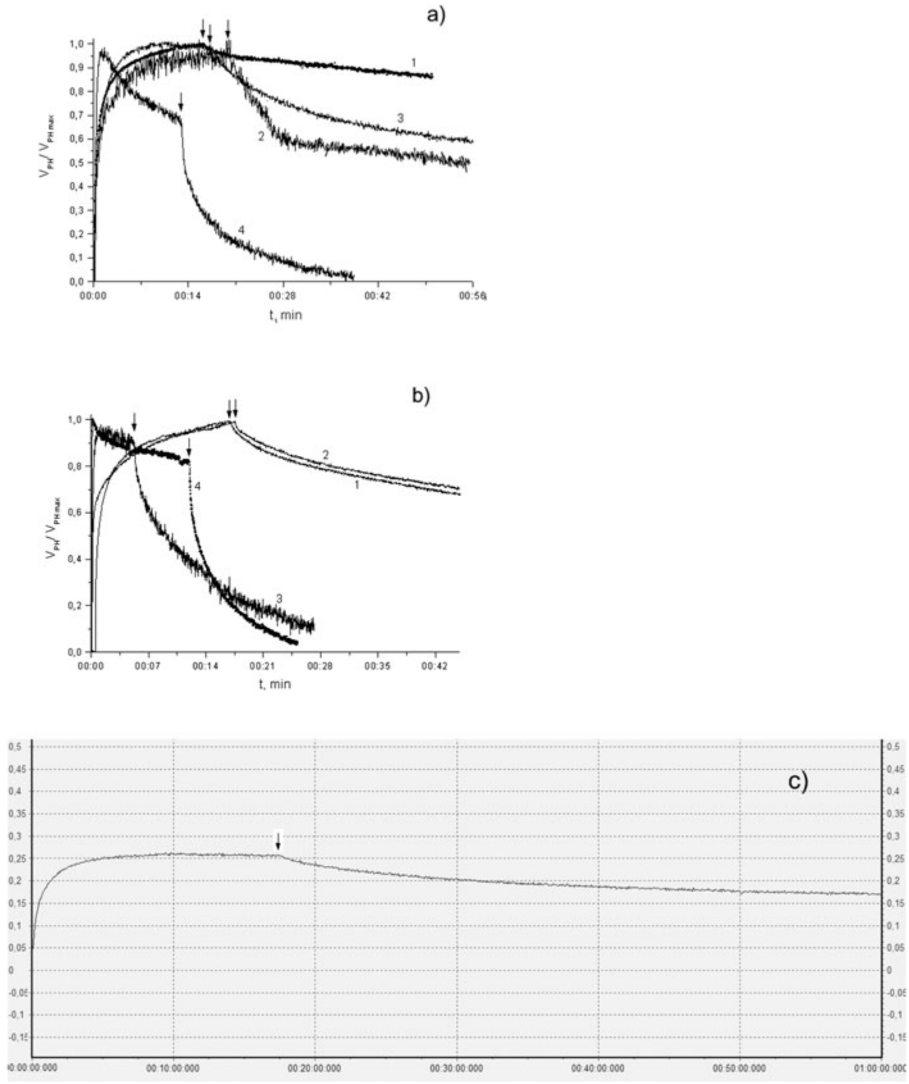


Figure 2. a) Normalized kinetics of the surface photovoltage growth and relaxation in the samples with a free surface PCFs based on PVA (curves 1, 2) and PS (curves 3, 4) doped with 1% of Rd (curves 1, 3) and wt.10 wt.% of Rd (curves 2, 4); b) Normalized kinetics of the surface photovoltage growth and relaxation in the samples with a free surface PCFs based on PVA (curves 1, 2) and PS (curves 3, 4) doped with 1% of Gn (curves 1, 3) and wt.10 wt.% of Gn (curves 2, 4); and c) Oscillogram of the surface photovoltage growth and relaxation kinetics in the sample with a free surface PCFs based on PVA doped with 1% of Gn (horizontal axis – time, min., vertical axis – photovoltage, V). In all these cases I = 50 W/m^2. The time moments of switching off the light illumination are shown by vertical arrows.

In accordance with the earlier accepted model concepts of photogeneration and transport of charge carriers in PCFs doped with organic dyes [1, 2, 5, 8, 9], the internal photoeffect in these systems is due to the formation of nonequilibrium charge carriers after absorption of light by dye molecules and transport of these carriers in the polymer matrix (We intentionally do not discuss in the present work in details possible mechanisms of the charge carriers photogeneration and transport in the investigated PCFs. Such photogeneration processes can involve electron or proton photoinduced transfer between monomeric dye molecules and the dyes aggregate species, charge transfer in the dye photoexcited states between dyes species and polymeric matrixes [1, 9, 11, 29]. The charge transport in such systems (for example in PVA based PCFs) can involve significant ionic component [9, 19, 26]. However, further study is required to establish the nature of discovered internal photoeffect in these systems).

It is suggested that observed in PCFs photovoltaic effect phenomenon is caused by the Dember effect, i.e. discovered effect has diffusion nature. This assumption was based on fact that observed effect appears in the range of strong light-absorption of high concentration dye-doped PCFs.

It is well known that the Dember (or photodiffusion) effect can be observed in semiconductors when the electrons and holes, (or, in the general case, electrons and holes, and ions of different types) have different mobilities and the light-illumination of the sample leads to a non-uniform distribution of charge carriers [17, 18]. The Dember effect (if the electrons and holes have different mobilities) is caused by the excitation light intensity and photogenerated charge (electron-hole) pairs concentration gradient in the film sample volume, respectively [18]. As a rule, the sign of the non-illuminated sample surface potential (with respect to illuminated surface) becomes the same as the charge sign of the photogenerated current carriers which have the larger mobility [17–18]. Under the exciting light illumination of the different sides of investigated samples the direction of this gradient becomes inverse. Therefore, under these conditions, the sign of the Dember's photovoltage also becomes inverse. To check the above-proposed assumption, the additional photovoltage measurements were carried out for symmetrical sandwich structure samples of investigated PCFs. Figure 3 illustrates the scheme and results of these experiments.

The Figure 3 (and the same photovoltage measurement results obtained for all other analogous symmetrical type sandwiched samples) shows that the sign of the photovoltage becomes inverse under exciting light illumination of the different sides of sandwiched samples. This fact confirms the correctness of our assumption about diffusion nature of the observed photovoltaic effect. In our systems case the potential of the non-illuminated (dark) surface becomes positive with respect to illuminated surface, because positive charged current carriers have the larger mobility. This confirms the p-type photoconductivity character of investigated PCFs [9, 16, 17]. The relatively slow photovoltaic response growth and very slow dark photovoltage relaxation kinetics character can be explained by a large influence of charge carriers capture on deep traps processes and low mobility of non-equilibrium charges in the PCFs. In the PVA based systems case can be also realized slow ionic polarization processes in the internal photoinduced Dember's electrical field in the corresponding composite films [9, 20, 21].

The dark conductivity current density j_d values for the samples based on PVA and PS without the dye and doped with dyes are practically the same. In the case of the PVA based PCFs the j_d kinetics under external electrical field exhibits the slow relaxation from initial to its stationary value with the characteristic time constants $\sim$ 30–100 s. Such behavior is typical for polymeric composites and dielectrics, which electroconductivity have ionic component [19, 20]. It was also established that all obtained PCFs exhibit photoconductivity under the light irradiation in the dyes absorption region. Moreover, in our PCFs systems we observed unusual anomalous negative photoconductivity effect. As an example, Figure 4 (curve 1) demonstrates the typical example of this effect in the case of PVA based

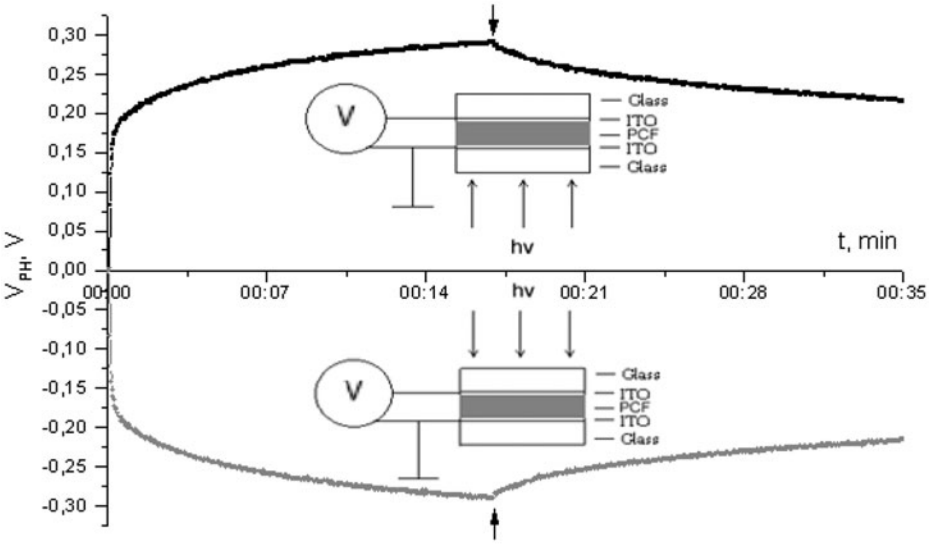


Figure 3. Normalized kinetics of the surface photovoltage growth and relaxation in the symmetrical ITO-PCF-ITO sandwiched sample based on PVA doped with 1 wt% of Gn under illumination of the different sides of sample by white light-emitting diode irradiation (I = 50 W/m^2), and the scheme of respective photovoltage measurements. The time moments of switching off the light illumination are shown by vertical arrows.

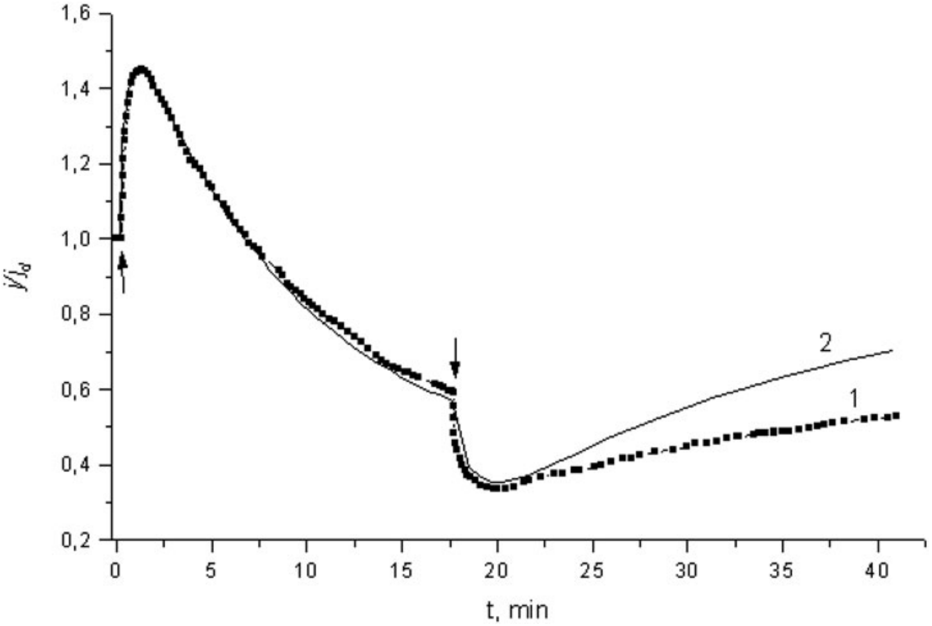


Figure 4. Dependences of j/j$_d$ on t (1) measured in ITO - PVA + 10 wt.% of Rd – Ag samples for E$_0$ = 7,5·10^7 V/m (1) and I = 50 W/m^2; and (2) calculated using the suggested phenomenological model. The times of switching light on and off are marked with arrows.

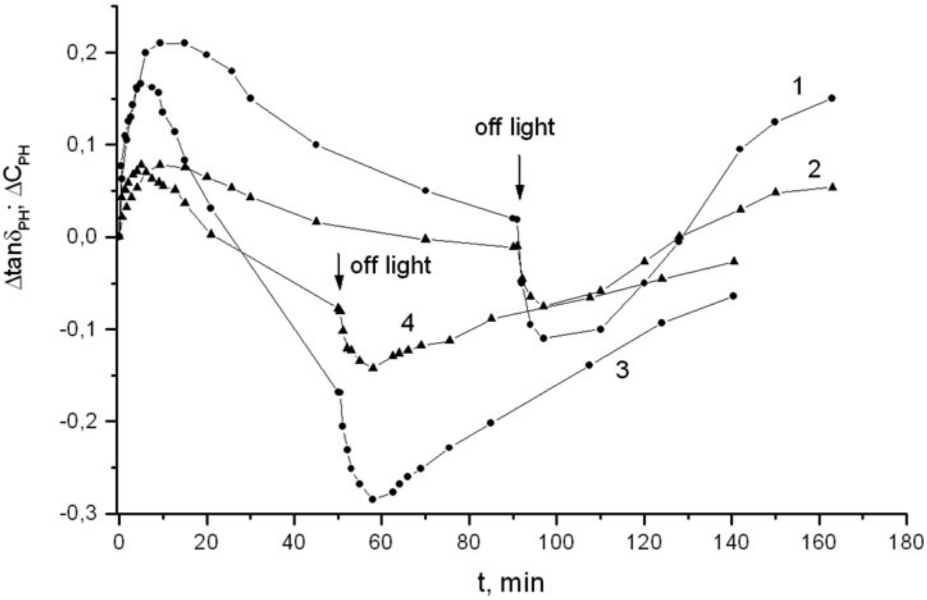


Figure 5. Dependences of Δtan δ_{PH} (1, 3) and ΔC_{PH} (2, 4) on t in the ITO – PCF – Ag sandwich structure samples based on PVA doped with 10 wt.% Rd (1, 2), and 10 wt.% Gn (3, 4). The time of switching off light is marked by arrows. Here, f = 1 kHz, I = 50 W/m^2.

sandwiched ITO - PCF – Ag samples doped with 10% wt. of Rd (U = 100 V). In these films, after switching on light, an increase in the conductivity current is initially observed, and then the j value decreases and becomes lower than the j_d value (Figure 4). The time of increasing the conductivity current to its maximum value after switching on light is much shorter than the time of relaxation of this current to the quasi-stationary value j and the time of its recovery to the j_d value after switching off light. Such kinetical character of j growth and relaxation occurs in all applied to investigated PCFs samples electric voltage range (in our experiments U = 5–120 V) and the value of j is practically independent of the polarity U. It is suggested that observed anomalous negative photoconductivity effect can be caused by the captured on deep traps (for example, on dyes associates species) photogenerated space charge effect on the charge carriers transport and their mobility [9, 13, 21–25].

To explain qualitatively the assumption made on the effect of capturing charge carriers on the conductivity current in PCF under study, we considered a simple phenomenological model of photogeneration, transport, and capture of nonequilibrium charge carriers. It was supposed in this simplified model that after switching on an external electric field with an intensity E_0 = U/L, an internal electric field with an intensity E(t) was established in the course of time t in the sample with the sandwiched structure. Before beginning of light irradiation, the density of the electric current is generally determined by ionic, hole, and electronic conductivities. This current density can be presented by a sum of the number (i) of the components for the particles having the electric charge Q_i, the concentration N_i, and the mobility μ_i:

$$j_d(t) = E(t) \times \sum_i Q_i N_i \mu_i, \tag{1}$$

Upon light irradiation, which produces photogeneration of carriers with the electric charge e equal to the elementary electric charge, the concentration n_e, and the mobility μ_e, the electric current density changes, and its dependence on t can be presented as follows:

$$j(t) = E(t) \times \left(\sum_i Q_i N_i \mu_i + e n_e \mu_e \right). \tag{2}$$

Due to capturing of photogenerated charge carriers by traps, sedentary sites with an electric charge q can appear in the bulk of PCF, and a change in the concentration (n_t) of these sites can initiate a change in the electric field intensity in PCF studied (as well as to a decrease of the charge carriers mobility [24, 25]). According to the works [13, 23, 25], this change can be presented as follows:

$$E(t) = E_0 - q\alpha\, n\mathrm{t}(t)/\varepsilon_0, \tag{3}$$

where ε_0 is the electric constant, α is the coefficient depending on electric characteristics of the medium, the electric field intensity, and temperature. With light irradiation, the concentrations $n_e(t)$ and $n_t(t)$ can be described by the following system of kinetic equations:

$$\begin{aligned} dn_e/dt &= G - (k_t + k_r + k_\eta)n_e + k_{-t}n_t, \\ dn_t/dt &= k_t n_e - k_{-t}n_t, \end{aligned} \tag{4}$$

where G is the efficiency of photogeneration of charge carriers, k_r is the recombination rate constant of these carriers, k_t is the rate constant of capturing of photogenerated charge carriers by traps, k_{-t} is the rate constant of release of charge carriers from traps, and k_η is the rate constant of exit of charge carriers to contacts. These constants and the values of Q_i, N_i, μ_i, μ_e, and α were the parameters of numerical simulation. With allowance for the initial conditions

$$n_e|_{t=0} = n_t|_{t=0} = 0, dn_e/dt(t = 0) = G, dn_t/dt(t = 0) = 0, \tag{5}$$

the solution of the system of kinetic equations (4) can be written as follows:

$$\begin{aligned} n_e = \frac{G}{K(k_r+k_\eta)}\Big\{ & K - \frac{1}{2}(k_t + k_{-t} - k_r - k_\eta + K)\exp\left(-(k_r + k_{-t} + k_t + k_\eta - K)\frac{t}{2}\right) \\ & + \frac{1}{2}(k_t + k_{-t} - k_r - k_\eta - K)\exp\left(-(k_r + k_{-t} + k_t + k_\eta + K)\frac{t}{2}\right)\Big\}, \end{aligned} \tag{6}$$

$$\begin{aligned} n_t = \frac{Gk_t}{k_{-t}K(k_r+k_\eta)}\Big\{ & K - \frac{1}{2}(k_r + k_{-t} + k_t + k_\eta + K)\exp\left(-(k_r + k_{-t} + k_t + k_\eta - K)\frac{t}{2}\right) \\ & + \frac{1}{2}(k_r + k_{-t} + k_t + k_\eta - K)\exp\left(-(k_r + k_{-t} + k_t + k_\eta + K)\frac{t}{2}\right)\Big\}. \end{aligned} \tag{7}$$

where $K = \{(k_r + k_{-t} + k_t + k_\eta)^2 - 4(k_r + k_\eta)k_{-t})\}^{1/2}$.

After switching off light (G = 0), the concentrations n_e and n_t can be described by the system of kinetic equations similar to (4), but for the new initial conditions:

$$n_e|_{t=0} = N_e;\ n_t|_{t=0} = N_t, \tag{8}$$

where N_e and N_t are the concentrations of mobile and captured charge carriers, respectively, at the moment of switching off light. The solution of the system of kinetic equations after switching off light is as follows:

$$\begin{aligned} n_e = \frac{k_{-t}N_t}{2k_tK}\Big\{&\big(k_t + k_{-t} - k_r - k_\eta + K\big)\exp\Big(-\big(k_r + k_{-t} + k_t + k_\eta - K\big)\frac{t}{2}\Big) \\ &+\big(k_r - k_{-t} - k_t + k_\eta + K\big)\exp\Big(-\big(k_r + k_{-t} + k_t + k_\eta + K\big)\frac{t}{2}\Big)\Big\}, \end{aligned} \tag{9}$$

$$\begin{aligned} n_t = \frac{N_t}{K}\Big\{&K + \frac{1}{2}\big(k_r + k_{-t} + k_t + k_\eta + K\big)\exp\Big(-\big(k_r + k_{-t} + k_t + k_\eta - K\big)\frac{t}{2}\Big) \\ &-\frac{1}{2}\big(k_r + k_{-t} + k_t + k_\eta - K\big)\exp\Big(-\big(k_r + k_{-t} + k_t + k_\eta + K\big)\frac{t}{2}\Big)\Big\}. \end{aligned} \tag{10}$$

Figure 4 (curve 2) shows the results of numerical simulation of the dependence of j/j_d on t after beginning of light irradiation at time $t = 0$ and subsequent switching off light at time $t = 16$ min for $U/L = 7.5 \times 10^7$ V/m. The calculation was performed under the assumption that $k_r, k_t, k_\eta > k_{-t}$; $k_t > k_r$. These results correlate with the experimental data and are consistent with our assumption that the accumulation of the space charge in FPC under study can be the reason for the effect of negative photoconductivity.

Our assumption also confirmed by photodielectric measurements data in the investigated PCFs [27, 28]. In agreement with changing j after switching light on and off, a change in the dielectric characteristics of PCF occurs (Figure 5 shows the respective examples for the PVA based PCFs doped with 10 wt.% of Rd and Gn).

It should be noted that all investigated PCFs exhibit a photodielectric effect [27, 28] under the photoexcitation in the dye absorption region. But in the PCFs doped with a low concentration of dyes this effect is not significant and its increases with the dye concentration in the PCFs increasing (for both investigated dyes). The decrease in j and $\tan\delta$, and C in the course of prolonged irradiation indicates the formation of a significant sedentary space charge in PCF due to the capture of nonequilibrium carriers onto deep traps [13, 21–23, 25–28]. After switching off light, these traps become free and the values of j_d, C and $\tan\delta$ are restored.

Conclusions

The new photoconductive PCFs based on non-photoconducting polyvinyl alcohol and polystyrene doped by various concentration of xanthene and triarylmethane type dyes have been prepared. Their photophysical, electrical, and spectral properties have been investigated. It was shown, that all studied composite films exhibit photovoltaic properties and anomalous negative photoconductivity effect. The Dember's nature of observed photovoltaic response was established. The features of photovoltaic, photodielectric effect and photoconductivity in the investigated PCFs, as well as the possible negative photoconductivity mechanism are discussed. The phenomenological model based on assumptions about the buildup of nonequilibrium space charge and its influence on electric conductivity of the investigated films is suggested.

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