Spectroscopic Study on Charge Generation in Bisazo-Based Layered Photoreceptor

S. Yokota

Digital Printing Division, Samsung Electronics Co., Ltd., Suwon City 443742, Korea

Abstract. A spectroscopic study was carried out on charge generation processes in a multilayered photoreceptor comprising a specific bisazo compound as charge generation material. Photoreceptors comprising a charge generation layer (CGL), composed of a bisazo compound dispersed in a binder resin, and a charge transport layer (CTL) incorporating an electron donor molecule in a binder resin, were treated as model photoreceptors for experimental and numerical analysis. Charge generation model, which had been previously described for a single layer organic photoconductor with charge generation predominately occurring at layer surface, was expanded and applied in a numerical study to a conventional dual layer system. A change in spectral response was observed as a result of variation in CGL thickness for both conventional dual layer and inverted dual layer (CTL overcoated with CGL) systems. These experimental results were compared with numerical simulations in the expanded charge generation model. Analytical results suggested that charge generation sites existed at CGL-CTL interface and that estimated energy diffusion length was much longer than exciton diffusion length reported for organic materials. Moreover, the author's proposed charge generation model showed superior agreement with experimental results compared to conventional bulk generation model. © 2010 Society for Imaging Science and Technology. [DOI: 10.2352/J.ImagingSci.Technol.2010.54.4.040505]

INTRODUCTION

Many studies on charge generation process in organic photoreceptors have been carried out, as it was regarded as a fundamental mechanism in the electrophotographic process. Those studies were successful to a certain extent, providing descriptions of, e.g., thermalization process in ion pair dissociation, adopting Onsager's model^{1,2} for both inorganic and organic photoreceptors, although the "thermalization process" concept was not altogether the same for inorganic and organic materials. In inorganic photoreceptors, it is usually regarded as an intrinsic process predominantly exhibiting dependence of thermalization length on photon energy, while in organic photoreceptors (OPCs), it is usually an extrinsic process without significant dependence of thermalization length on photon energy.

Azo compounds,³ as well as phthalocyanine compounds, are widely used substances as charge generation materials (CGMs) in commercial photoreceptors. In general, azo compounds have a larger ionization potential and wider band gap than phthalocyanine compounds so that they are usually used in photoreceptors that are employed in engines

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using visible light for image exposure, such as analog copy machines or laser printers that employ short wavelength laser scanners. The first bisazo pigment to find application as CGM in an electrophotographic system was Chlorodiane Blue^{4,5} in a layered OPC for copy machines produced by IBM.

Succeeding applications as well as studies on charge generation process in azo based photoreceptors were mainly carried out by several Japanese companies. Umeda et al.⁶ and Niimi et al.⁷ studied charge generation in layered OPCs with specific azo pigments as CGM and an electron donor compound as charge transport material (CTM). They proposed that photoexcitation occurred in bulk CGL and that the energy is transferred into CGM particles by exciton diffusion. Dissociation of electron-hole pairs occurs to free carriers only at the surface of CGM particles in contact with CTM molecules, which presupposes penetration of CTM molecules into CGL during the coating process. This generation model represents the generally accepted interpretation of charge generation process in multilayer type photoreceptors that utilize azo compounds as CGM.

However, in proposing charge generation in bulk CGL, one must inevitably consider effects of energy dissipation due to Langevin recombination, where recombination rate depends directly on carrier density.^{8,9} This produces some difficulty in interpreting experimental results for some dual layer (DL) photoreceptors comprising specific azo^{10,11} or phthalocyanine¹² compounds that exhibit uniquely high quantum efficiency with a very small dependence on photon energy.

On other hand, Bilke et al.¹³ studied charge generation in dual layer photoreceptors comprising a bisazo pigment based CGL and a liquid crystalline donor compound based CTL. They suggested that charge generation occurred at CGL surface with assistance of long range exciton diffusion. It is interesting that they proposed and experimentally demonstrated charge generation at the interlayer interface although it raises some doubt as to how the exciton is transferred for such a long range over multiple bisazo particles embedded in an insulating polymer.

In previous work,^{14,15} we proposed a new model of charge generation in single layer (SL) OPCs. We suggested photoexcitation in the bulk of the layer, which is then followed by charge separation only at the layer surface assisted by energy diffusion in a similar manner as the model adopted for exciton diffusion for a thin merocyanine film by

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Gosh et al.¹⁶ Although architecture of SL system in those earlier studies is not exactly identical to that of dual layer (DL) photoreceptor systems, the energy diffusion concept adopted is essential, and applicable in general to all OPC system.

In this article, we will expand our proposed model to a more general multilayered OPC system comprising CGL and CTL. Main experimental focus of this study is to observe dependence of spectral response on CGL thickness and on direction of incident light, toward or against CGL, in which a specific bisazo pigment is employed as CGM. Next, we will discuss adaptability of the model to these experimental results. Moreover, comparison of the applicability of our generation model and of the conventional bulk generation model will be discussed.

EXPANSION OF CHARGE GENERATION MODEL

As shown in our previous paper,¹³ we assume that charge generation occurs only at layer surface, in order to explain spectral characteristics of both positively and negatively charged sensitivities in a positive carrier dominating SL system. A characteristic of that system was that the whole layer has both functions of charge generation and charge transport simultaneously. In the case of a DL system, it is obvious that CGL must have a charge transport function as well as a charge generation function in order to allow generated carriers to pass through CGL itself although it is conventionally assumed that these functions of charge generation and charge transport are separated into CGL and CTL. Consequently, it may be reasonable that we treat charge generation in CGL in a DL system in the same manner carried out for that of an SL system.

Generally, bisazo pigment is known to be an n type semiconductor,¹⁷ which means that the majority carriers in CGL comprising bisazo pigment would be electrons. Contrarily, majority carriers in CTL comprising a solid solution of electron donor compound in binder resin would be holes. Supposing conjunction of two layers with thus opposite polarities, we propose that the charge generation site must be located at the interface of two layers when our surface generation model is adopted.

When light exposure with N_0 photons occurs toward the surface of a CGL with thickness *l*, photon number N(x)at depth position *x* is expressed by the following equation where light absorption coefficient is α :

$$N(x) = N_0 \exp(-\alpha x). \tag{1}$$

In consideration of exponential decay due to diffusion from position x to the charge generation site at the layer surface, charge generation resulting from photoexcitation at position x can be expressed as follows:

$$\frac{dn}{dx} = \phi \left(-\frac{dN(x)}{dx} \right) \exp \left(-\frac{X}{L} \right) = \phi N_0 \alpha \exp(-\alpha x)$$
$$\times \exp(-\beta X), \tag{2}$$

where *n* is carrier number, ϕ is quantum efficiency, β is

Figure 1. Schematic illustrations of OPC profile. (a) DL structure and (b) IDL structure.

reciprocal of diffusion length *L*, and *X* denotes distance from charge generation site to position *x*, that is, X=x when charge generation occurs at surface of incidence of light exposure, and X=l-x for the opposite surface.

We can obtain total carrier number, which should be proportional to sensitivity, by integrating this equation over entire layer thickness as follows:

$$n = \int_{0}^{l} \phi N_0 \alpha \exp(-\alpha x) \exp(-\beta X) dx.$$
 (3)

In addition, with respect to the structure of multilayered OPC, we can suppose two kinds of layer systems. One is the more popular DL system in which CTL is the top layer covering CGL; the other one is the positive charging type of inverted dual layer (IDL) system, having CGL as top layer. These constructions are illustrated in Figures 1(a) and 1(b). Interface of CGL and CTL as charge generation sites exist on the top surface of CGL in case of DL system and on the bottom of CGL in the case of IDL system. Thus, parameter *X* in Eq. (3) should be X=x for DL system and X=l-x for IDL system.

In this article, we define sensitivity as the rate of discharge of surface potential due to light exposure. From integration of Eq. (3) for CGL thickness, we can get model equations for sensitivities of both different layered systems as follows:

$$S_{DL} = \frac{e\phi_{DL}P_0\alpha}{Ch\nu(\beta+\alpha)} \{1 - \exp(-\beta l - \alpha l)\}$$
(4)

for DL system and

$$S_{IDL} = \frac{e\phi_{IDL}P_0\alpha}{Ch\nu(\beta-\alpha)} \{\exp(-\alpha l) - \exp(-\beta l)\}$$
(5)

for IDL system, where *C* is capacitance of total construction, including CTL, and P_0 is intensity of incident light.

EXPERIMENTAL

OPC Samples

Bisazo pigment whose structure is shown in Figure $2(a)^{18}$ was dispersed in polyvinyl butyral resin in 1:1 weight ratio with mixed solvent of seven parts of methyl alcohol and three parts of ethyl acetate by a ball mill. The prepared dispersion liquid was coated on anodized aluminum drums by a ring coating method to form CGL. In order to compare



Figure 2. Chemical structures of (a) Bisazo compound as CGM and (b) Stilbene compound as CTM.

effects of CGL thickness, two samples with thin and thick CGLs were prepared in same way. We estimated thicknesses by relative light absorption against reference sample as 0.4 and 1.6 μ m, respectively.

On the CGL, a solution of a stilbene compound¹⁹ [4-N,N-bis(4-methyphenyl)amino- α -phenylstilbene, structure shown in Fig. 2(b)] and polycarbonate resin in 4:6 weight ratio dissolved in tetrahydrofran was coated similarly to form a 17- μ m-thick CTL after drying.

Additionally, we prepared IDL samples with two different CGL thicknesses by coating CGL on 17 μ m CTL using same coating liquids as for DL samples, as illustrated in Fig. 1. In this case, estimated thickness of the thinner CGL was 0.4 μ m and the thicker one was 1.0 μ m.

Absorbance Spectrum

An approximately 1.0 μ m thick CGL film was formed on a transparent PET sheet by spin coating and drying using the same CGL solution used for photoreceptors preparation. CTL film on another PET sheet was also prepared in same way using corresponding CTL solution. UV-visible spectra of CGL and CTL films thus prepared were directly measured with a UV-visible spectrometer, Model 8453 from Agilent Technologies.

Spectral Sensitivities

Spectral sensitivities of both DL and IDL samples were measured using a photoelectric drum tester, Cynthia 91 of GENTEC Co., Japan, in the same manner as described in our previous paper.¹⁴ Photoinduced discharge curves (PIDCs) at each photosensitive wavelength were measured in static mode using an electrostatic voltmeter with a transparent probe while applying continuous exposure of 2 μ W/cm² monochromatic light on OPC surface, which had been charged to 500 V in absolute value. Sensitivity *S* was defined as initial rate of light decay of surface potential corrected for dark decay as follows:

$$S = \left| \frac{dV_D}{dt} - \frac{dV_L}{dt} \right|, \tag{6}$$



Figure 3. Absorbance spectra of (a) CGL and (b) CTL films coated on PET sheets.



Figure 4. Experimental spectral sensitivities according to Eq. (6) of DL photoreceptors: (a) 1.6- μ m-thick CGL and (b) 0.4- μ m-thick CGL.

where V_D and V_L mean dark and light potentials just before and after start of light exposure, respectively.

EXPERIMENTAL RESULTS

Absorbance Spectra

Figure 3 shows absorbance spectra of CGL and CTL films coated on transparent PET sheets. Bisazo pigment exhibited strong absorption from the UV region to around 600 nm, and substantially no absorption at wavelengths longer than 700 nm. Peak absorption (λ_{max}) was found at around 530 nm.

On the other hand, CTL was transparent over most of the visible region, but exhibited prominent absorption at wavelengths shorter than 450 nm.

Spectral Sensitivities

Figure 4 shows spectral sensitivities of DL samples with thick and thin CGLs, where a remarkable increase in sensitivity and a shift of sensitivity peak to longer wavelength can be seen with increasing CGL thickness. Unlike absorbance spectra of CGL, sensitivity shows a rapid drop at wavelengths shorter than 450 nm, where absorption of CTL appears. Results for IDL samples shown in Figure 5 also show a similar sensitivity increase and shift of peak to longer wavelength on increasing CGL thickness, along with the rapid sensitivity drop at shorter wavelengths.



Figure 5. Experimental spectral sensitivities according to Eq. (6) of IDL photoreceptors: (a) 1.0- μ m-thick CGL and (b) 0.4- μ m-thick CGL.

DISCUSSION

Determination of Diffusion Length

In respect to spectral sensitivities, it was shown that not only absolute value but also spectral dependence of sensitivity changed on varying CGL thickness for both DL and IDL photoreceptors, where sensitivity spectra became broader and the peak sensitivity shifted to longer wavelengths with increasing CGL thickness. If our proposed generation model is correct, these phenomena should be traced numerically using our model Eqs. (4) and (5).

In order to perform accurate simulation of spectral sensitivity using our model equations, the key parameter would be diffusion length $L = \beta^{-1}$ the same as in the case of an SL system. Hence, it is very important how accurately we can estimate this value for discussion hereafter. We can make use of the analytical result that spectral dependence of sensitivity depends only on variables α , β , and l as shown in model Eqs. (4) and (5), where values of α and l are already known from experimental results. Therefore, we can estimate β by fitting calculated curves to experimental curves of spectral sensitivity using experimental data for α and l.

Before calculation, we should further consider effects of light reflection from the substrate on sensitivity, as we used bright anodized aluminum as the substrate. We can suppose that this contribution of reflected light to sensitivity is roughly proportional to transmittance of CGL, $\exp(-\alpha l)$. That is, apparent sensitivity S_A would be larger than natural sensitivity S_0 and expressed in following equation, where *R* is reflectivity index of the substrate:

$$S_A = S_0 \{ 1 + R \exp(-\alpha l) \}.$$
 (7)

This equation suggests that observed sensitivity becomes closer to natural sensitivity when CGL with a larger absorption coefficient is employed. Therefore, we will use data for the thicker CGL sample, that is, DL sample with 1.6- μ m-thick CGL, for estimating β .

Supposing quantum efficiency ϕ_{DL} to be independent of wavelength, we can simulate spectral sensitivity for any proposed β value using absorption coefficient data obtained from the absorbance spectrum for 1.0 μ m thick CGL as



Figure 6. Simulated spectral sensitivities of DL photoreceptors according to Eq. (4) with 1.6- μ m-thick CGL changing β value from 0 to 2 × 10⁴ by 1 × 10³ steps. Bold line shows optimal curve corresponding to experimental values denoted as open circles.

shown in Fig. 3. Fig. 5 shows spectral sensitivities thus calculated from Eq. (4) by varying the value of β from 0 to 2 $\times 10^4$ cm⁻¹ substituting CGL thickness *l* as 1.6×10^4 cm, the experimental value for the thicker CGL. Figure 6 simultaneously shows experimental data of DL OPC with $1.6-\mu$ m-thick CGL adjusting the relative magnitude so as to optimally fit to the calculated curve.

We can neglect experimental data at wavelengths shorter than 450 nm which show a large drop from the theoretical curve, since this result is obviously due to light absorption by CTL as shown in Fig. 3. In same way, we can also neglect sensitivity drops at wavelengths longer than 650 nm. Observed absorption spectra may include many factors, such as surface reflection or scattering of incident light, other than the natural absorption of the material, and sensitivity is only result of this natural absorption. Considering experimental results showing almost no sensitivity at 700 nm, we suppose that actual light absorption in this spectral region by this pigment to be much smaller than observed.

Comparing curves of Fig. 6 with data for the thicker CGL in Fig. 4, we infer that spectral sensitivity of thick CGL approximately fits to the curve calculated for $\beta = 2 \times 10^3$ cm⁻¹ over the wavelength range 450–650 nm. Therefore, we can estimate β to be about 2×10^3 cm⁻¹; that is, diffusion length *L* is approximately 5 μ m, which is coincidentally equivalent to the value estimated for an SL OPC in our prior study,¹⁴ and much longer than exciton diffusion lengths of 130–150 nm for a specific azo based CGL proposed by Bilke et al.¹³ or even than actual CGL thickness.

Although our diffusion length was estimated in a rough approximation, one order of magnitude difference from that by Bilke et al. seems to be essential. One possible reason for this difference may be the difference in materials employed in each experiment. For example, quantum efficiencies of photoreceptors of Bilke et al. were smaller by one or two orders magnitude than those of our experiment, and such a fundamental difference may cause a difference in diffusion length as well, considering that the diffusion length of a deposited merocyanine film was only 60 Å.¹⁶



Figure 7. Simulated spectral sensitivities of DL photoreceptors with various CGL thicknesses according to Eq. (4). Bold lines show those of 0.4-µm- and 1.6-µm-thick CGLs corresponding to experimental photoreceptors. Open and solid circles show experimental results for photoreceptors with 0.4-µm- and 1.6-µm-thick CGLs, respectively.



Figure 8. Simulated spectral sensitivities of IDL photoreceptors for various CGL thicknesses according to Eq. (5). Bold lines show those of 0.4- μ m-and 1.0- μ m-thick CGLs corresponding to experimental photoreceptors. open and solid circles show experimental results for photoreceptors of 0.4- μ m- and 1.0- μ m-thick CGLs, respectively.

Simulation and Comparison with Experimental Data

We can simulate thickness dependence of spectral sensitivity using the estimated β value.

Figure 7 shows calculated spectral sensitivities using fixed β value and various CGL thicknesses from 0.1 to 2.0 μ m in model Eq. (4). A clear increase in sensitivity and a sensitivity shift to longer wavelength can be seen on increase in CGL thickness, the same as the experimental result shown in Fig. 4. Similarly, we can simulate spectral sensitivity of IDL photoreceptors using Eq. (5) and absorption spectral data as shown in Figure 8.

Unlike the case of SL OPCs, spectral dependence of IDL OPC is apparently similar to that of DL OPC. It shows a similar tendency of sensitivity increase and wavelength shift, the same as simulated for DL OPC, and the experimental result shown in Fig. 5 corresponds well with it. However, it shows a significant difference from DL insofar as sensitivity starts decreasing at wavelengths of large light absorbance when CGL becomes thicker.

Figs. 7 and 8 also show experimental spectral sensitivities as open and solid circles, adjusting ϕ values so as to best fit data for thicker CGL samples to theoretical curves over the wavelength range from 450 to 650 nm, where capacitance C was calculated from total layer thickness and dielectric constant assumed to be 3.0 uniformly for the whole layer. As shown in these figures, experimental results show good correspondence to theoretical curves in both DL and IDL systems. On other hand, it must be noted that experimental values for thinner CGL samples are larger than calculated curves. This deviation of experimental values from theoretical ones may be proof of increased sensitivity due to penetration of light reflected from the substrate as mentioned above, which will be more significant when a thin CGL with higher transmittance is used.

Quantum efficiencies for both DL and IDL systems, which were estimated by fitting experimental data to simulation curves, are $\phi_{DL}=0.17$ and $\phi_{IDL}=0.11$, respectively. These values inevitably include some uncertainty, considering deviation of experimental sensitivity values from simulation curves. But the difference between 0.17 and 0.11 may be large enough to be regarded as significant. Thus, we infer that quantum efficiency of a DL system is larger than that of an IDL system. This result is reasonable because solvent for CTL is also a good solvent for CGL binder so that penetration of CTM molecules into CGL may easily occur in a DL system when coating the CTL, as suggested by Niimi et al.⁷ Although Niimi et al. explained this sensitization as result of increase in number of charge generation sites in bulk where contact of CGM particles and CTM molecules occurred, we can interpret it differently adopting our surface generation model, where penetrated CTM molecules generate hole mobility in the CGL allowing additional charge generation at the opposite side surface of the CGL, and apparent quantum efficiency increases as a result. On other hand, since solvent for CGL is not such a good solvent for CTL binder penetration of CTM molecule into CGL of IDL system may be much smaller than in DL system, so that we see a difference in quantum efficiencies as a result of the coating order of the layers, after all. Another possible interpretation of this sensitivity difference may be that contact of bisazo pigment with CTL solvent when coating CTL on CGL caused some morphological change in the pigment and consequent increased quantum efficiency, as suggested by Pacansky et al.²⁰ although no apparent difference in absorption spectra with layer structure could be found in this experiment.

Comparison with Bulk Generation Model

Conventional bulk generation model proposes ion pair dissociation proportional to total photon number absorbed in CGL. Therefore, neglecting Langevin recombination, we simply express sensitivities for both DL and IDL photoreceptors in a single equation as follows:

$$S_{con} = \frac{e\phi P_0}{Ch\nu} \{1 - \exp(-\alpha l)\}.$$
(8)

Spectral sensitivity simulation using this equation is easily carried out using absorbance spectral data for CGL shown in Fig. 4. In same manner as simulations shown in



Figure 9. Simulated spectral sensitivities for various CGL thicknesses by bulk generation model according to Eq. (8). Bold lines show those of 0.4- μ m- and 1.6- μ m-thick CGLs corresponding to experimental DL photoreceptors. Dotted lines show those by our proposed model for DL photoreceptors.

Figs. 7 and 8, simulated spectral sensitivities for the bulk generation model with various CGL thicknesses are shown in Figure 9, where simulation curves by our suggested DL model for 0.4- μ m- and 1.6- μ m-thick CGLs are drawn simultaneously for comparison.

It is obvious that spectral curves of both models are almost identical when CGL thickness is small but become different with increasing CGL thickness. The main difference is that the simulation curve for the conventional model exhibits a sensitivity peak at rather longer wavelength than that of our DL model and shows quick saturation at wavelengths around 450 to 580 nm, corresponding to the region of large light absorption. Although it does not seem impossible to explain experimental results for DL system using these simulation curves based on bulk generation model, our generation model seems to be more natural and fit better to the experimental data than bulk generation model.

On other hand, the conventional model would not suggest a sensitivity decrease at wavelengths of large light absorption on increasing CGL thickness of the IDL system as suggested in our proposed model. Bilke et al.¹³ already showed experimentally that a significant decrease in sensitivity appeared at wavelengths of large light absorption for a DL system with thick azo based CGL when light was exposed from the electrode side, substantially equivalent to our IDL system with surface light exposure. This is proof for validity of our charge generation model, differentiating it from the conventional model.

CONCLUSION

A charge generation model assuming energy diffusion from bulk by photoexcitation and charge generation at a layer interface, which had been proposed for SL OPC, was expanded and applied to multilayered photoreceptors comprising a bisazo pigment as CGM. Experiments using spectroscopic methods were carried out for both DL and IDL photoreceptors with various CGL thicknesses, and results were compared with those of numerical analysis adopting the expanded generation model. Although actual processes may include many supplemental factors such as contribution from light reflected from substrate or charge generation at the opposite surface owing to additional hole mobility in CGL, analysis here was carried out on a very simplified model, neglecting those factors.

Conclusions can be summarized as follows:

(i) Surface charge generation model proposed for a SL system was modified and applied to multilayered systems showing good agreement of experimental results and numerical analysis without any contradiction.

(ii) Photoexcitation energy due to light absorption in bulk CGL is transferred by energy diffusion into CGL, and charge generation takes place only at interface of CGL/CTL in both DL and IDL systems.

(iii) Estimated energy diffusion length in CGL was about 5 μ m corresponding to that of SL OPC, and much longer than those in prior studies on exciton diffusion.^{13,16} This long diffusion length may allow good energy efficiency, which will explain extremely high quantum efficiency of some layered photoreceptors.^{10,12}

(iv) Quantum efficiency estimated from modeling the DL system was larger than that for the IDL system, which we supposed was due to increase in hole mobility in CGL by CTM penetration into CGL.

(v) In comparison with bulk generation model, our proposed generation model showed superiority in matching of numerical simulation results with observed phenomena.

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