

A Study on Photoreceptor by Using the Effect of Additives

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We have been studied photosensitization mechanism's additive effect, of perylene 3,4,9,10-tetracarboxyldiimide and X-phthalocyanine (charge generation materials), using the photochemical and photoelectrochemical approach. It was found that the photoreceptor on the excited state reacts with metal oxide, which creates the charge transfer on the interface of SnO₂/electrolyte. In the electrode (X5P1) made of five X-phthalocyanine and single perylene 3,4,9,10-tetracarboxyldiimide layers, the cathodic photocurrent of X-phthalocyanine in the 400-600 nm region was increased by the addition of perylene 3,4,9,10-tetracarboxyldiimide. The maximum wavelength of fluorescence of perylene 3,4,9,10-tetracarboxyldiimide showed no dependence on the temperature. The addition of 4-dibenzylamino-2-methylbenzaldehyde diphenylhydrazone known as charge transport material was represented as decreasing photocurrent for X-phthalocyanine and perylene 3,4,9,10-tetracarboxyldiimide, respectively. In the electrode (X1P1) made of single X-phthalocyanine and single perylene 3,4,9,10-tetracarboxyldiimide layers, an anodic photocurrent of about 10.5 nA was generated by addition of hydroquinone at 550 nm. And the characteristic of photoinduced discharge was shown to decrease by a factor of 5 and the speed of dark decay was increased by a factor of 1.2.

Keywords : Photoreceptor, LB thin film, Photocurrent, Perylene derivatives.

Introduction

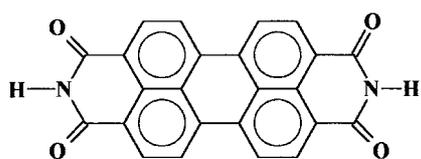
Studies of the electrochemical photosensitization mechanism of electron transfer through semiconductor interface have proceeded broadly,¹⁻⁴ and studies of the transformation of solar energy into effective energy have also been carried out widely.⁵⁻¹⁴ Hauffe argues that orange II and rhodamine B have sensitizing properties, photo-excited orange II transfers energy to rhodamine B, and the transferred electron is injected into metal oxide semiconductor.¹⁵ Also the research group of Tanaka found that it is possible to expand into the visible light region by adsorbing p type and n type dyes together into a metal oxide material.^{16,17} There has been reported that a high photocurrent is obtained when metallic atoms of a semiconductor are strongly combined with oxygen atom of dyes, such as rose bengal or xanthene, due to the high speed of transmitting electrons and the photocurrent is proportional to the amount of adsorbed dyes and depends on the pH.^{18,19} One of the difficult problems of photoconductor-electrolyte cell is the instability of the semiconductor electrode and electrolyte. Dwald study the electrochemistry of ZnO single crystals.²⁰ To obtain a photocurrent through a photoelectrochemical cell requires electron transfer between SnO₂/electrolyte. The increase in oxidation photocurrent occurs as a result of more electrons injected into the conduction band of SnO₂. The study of the fluorescence of a photoconductor is very valuable in explaining the processing route of charge generation.²¹ Menzel studied fluorescence and its quenching of metal free phthalocyanine and the excited singlet state, reporting medium electron transfer.²² The photoelectric properties of perylene 3,4,9,10-tetracarboxyldiimide derivatives in cyclic conjugated molecule can control the broad area by appending electron donor and electron accep-

tor.²³⁻²⁶ It is known that such excitation of photoconductor produces exciton, and the photogeneration mechanism of an organic photoconductor includes the reaction of electron transfer between exciton and electron donor or electron acceptor.²⁷⁻²⁹ However, information on the relation between their molecular structure and photosensitization mechanism is not enough. Therefore, the purpose of the present study is to analyze the photosensitization effect of X-phthalocyanine and perylene 3,4,9,10-tetracarboxyldiimide as charge generation material by investigating photochemical and photoelectric properties of methylviologen, hydroquinone and 4-dibenzylamino-2-methylbenzaldehyde diphenylhydrazone.

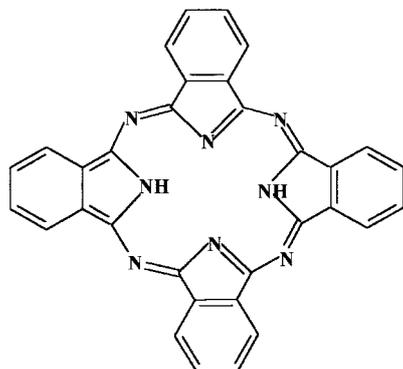
Experimental Section

Materials. We use specific grade reagent of sodium nitrate from Shinyo pure chemicals as electrolyte, and the 4-dibenzylamino-2-methylbenzaldehyde diphenylhydrazone was synthesized in 3-steps. We also use specific grade reagent from Aldrich as methylviologen dichloride hydrate of electron acceptor and X-phthalocyanine from Fuji Xerox Co. Perylene 3,4,9,10-tetracarboxyldiimide was obtained from Tokyo Kasei, we also used specific grade reagent in this case. The structural formula of these materials is shown in Figure 1.

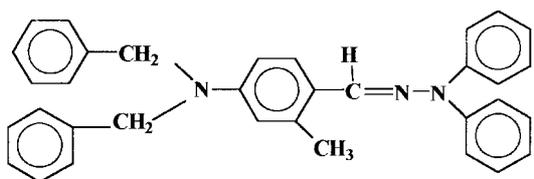
Instruments. We measured the absorption property of X-phthalocyanine and perylene 3,4,9,10-tetracarboxyldiimide, using the UV-Vis. spectrophotometer (Hewlett packard 8452A) with photodiode array. The construction of thin film was accomplished with Langmuir-Blodgett film preparation equipment from Apex Co.. To measure the photoinduced discharge property, we use a high voltage surface potentialmeter with EF-meter (electrophotometer).



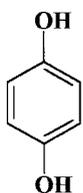
peryene 3,4,9,10-tetracarboxyldiimide



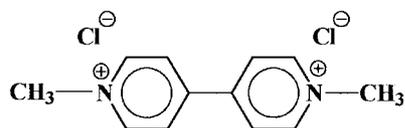
X-phthalocyanine



4-dibenzylamino-2-methyl benzaldehyde diphenyl hydrazone



hydroquinone



methyl viologene dichloride

Figure 1. Structural formula of materials.

The measurement of fluorescence. The compound of X-phthalocyanine and perylene 3,4,9,10-tetracarboxyldiimide on ITO glass electrode by the LB method were used. For the wavelength, we used a 514.5 nm Ar-ion laser (Spectra physics, 2016), placing the interference filter and cut-off filter (Corning 7-56) directly in front of the laser to eliminate unnecessary waves, and a light chopper (Rofin, 7500) was used to intermit excitation light. The strength of the laser beam was measured by a photoquantity measurement instrument (Spectra physics 404), and signals were detected by a GaAs detector.

Preparation of LB thin film. We chose a 5 cm × 2.5 cm of hydrophilic ITO glass (50 Ω/cm²). We used deionized water (18.2 mΩ/cm²) distilled by millipore/milli-Q deionized water equipment, with the temperature of the trough maintained at 25 °C. We started the experiment of isotherm, waiting 20 minutes after a drop of X-phthalocyanine and

peryene 3,4,9,10-tetracarboxyldiimide fell on the water, respectively. We made a thin film of Z-type by preserving the pressure on the surface at 20 mN/cm² on X-phthalocyanine, 10 mN/cm² on perylene 3,4,9,10-tetracarboxyldiimide, maintaining feedback control and dipper speed in 5 mm/min.

The measurement of photocurrent. The photoelectrochemical cell consisted of a three electrode systems with Ag/AgCl as the reference electrode and platinum wire as the auxiliary electrode. We used thin film made by the LB method on hydrophilic ITO glass as a working electrode. We used a sheet of pyrex as a photoelectrochemical cell and 1.0 M sodium nitrate as an electrolyte. The concentration of oxidant-reductant was 5 × 10⁻⁴ M. We used an electrolyte solution without eliminating dissolved oxygen. A 1 kW-halogen lamp was used as the light source and monochromatic light was obtained by using an interference filter.

Preparation of photoreceptor. We used a mylar sheet with a 42 μm Aluminum-coated surface on a PET film as a substrate of the photoreceptor. We made the thickness of the charge generation layer by using a wire bar of 0.2 μm and preserved that of the charge transfer layer as 5 μm. Thickness measurement equipment (MKC, A2002S) was used to measure the thickness of the sample. X-Phthalocyanine of 0.44 g was dispersed with 0.32 g of polycarbonate in 19.24 ml of chloroform for 3 hours. The concentration of perylene 3,4,9,10-tetracarboxyldiimide was made in the proportion of 0.01, 0.5, 1.0, 2.0 weight for X-phthalocyanine. In the charge transfer layer, 8.0 g (0.017 mole) of 4-dibenzylamino-2-methylbenzaldehyde diphenylhydrazone and 10.0 g (10 wt%) of polycarbonate were dissolved in 82 mL of chloroform. The cross section of the bilayer photoreceptor

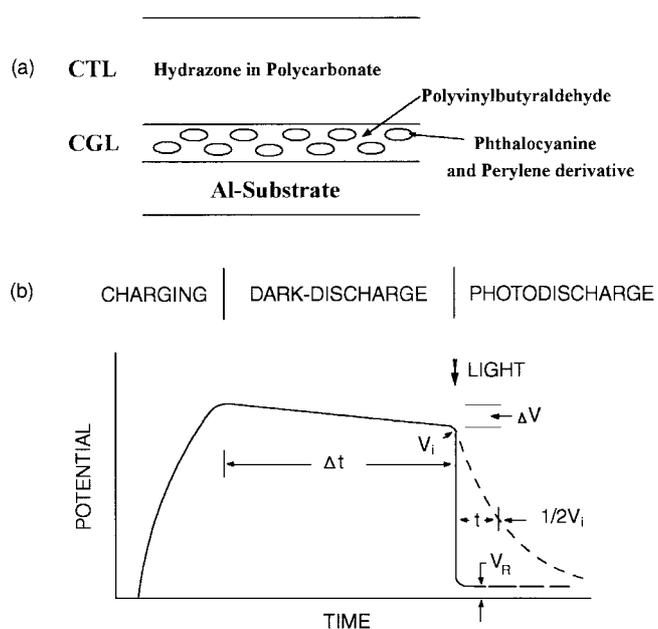


Figure 2. (a) A cross section of a bilayer photoreceptor device; (b) Schematics of photoinduced discharge curves, —, photoinduced discharge by an intense erase light; ---, photoinduced discharge by a monochromatic light at 600 or 800 nm.

film and the outline of the photoinduced discharge curve are shown in Figure 2. The sample was corona discharged at -6.0 kV, xenon lamp of 0.1 mW in light intensity was used as the light source.

Results and Discussion

Characteristics of the absorption spectra. Choosing a solvent that preserves crystal form is very important because in the crystal structure of phthalocyanine, the stacking arrangement is changed due to the combination of molecules by Vanderwaals force. The molecule of phthalocyanine has D_{4h} symmetrical characteristics approximately, and the orbit of π has the opposite function against the mirror image operation of the molecular symmetric plane. These functions include $e_g, a_{1u}, a_{2u}, b_{1u}, b_{2u}, a_{1u}$ is HOMO, and e_g becomes LUMO. A transition from a_{1u} to e_g ($\pi-\pi^*$) is the absorption of Q band, and a transition from a_{2u} to e_g ($\pi-\pi^*$) is related with the Soret band (B band). Figure 3(a) shows the absorption spectrum of phthalocyanine monomer. The division in 1-chloronaphthalene at 665 nm and 698 nm is due to the magnitude of the distortion from the D_{4h} symmetrical characteristics, and in the solid state it is due to the different circumstances of each molecule by molecular interaction in crystalline, which differs from the absorption spectrum in the solution state.²⁹ We observed the absorption peak of phthalocyanine monomer dissolved in 1-chloronaphthalene. In the case of phthalocyanine monomer dissolved in 2-propanol, we obtained the absorption peak while keeping the structure of X form on. We ascertained that the polarity of the solvent, affected the crystal structure of the solid, and a specific crystal structure could be achieved by controlling the solvent. Figure 4 shows an absorption spectrum in perylene 3,4,9,10-tetracarboxyldiimide solution. Strong absorption in $c\text{-H}_2\text{SO}_4$ was observed at 595 nm and 551.5 nm, which means this is similar to the absorption spectrum of the

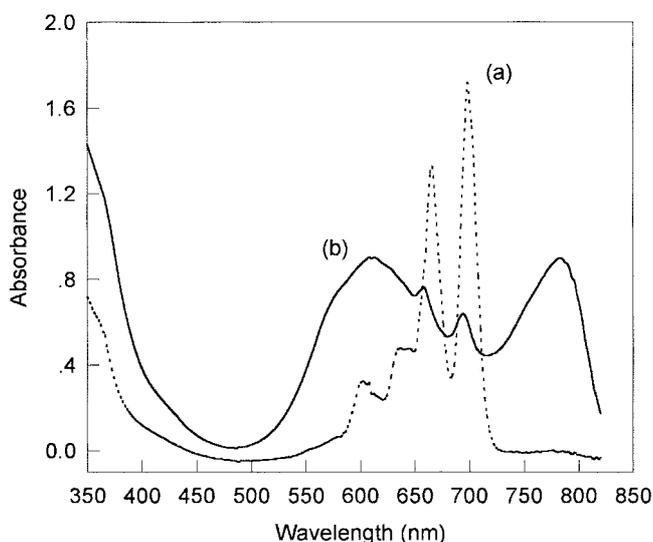


Figure 3. Absorption spectra of X-phthalocyanine. (a) 1-chloronaphthalene; (b) 2-propanol.

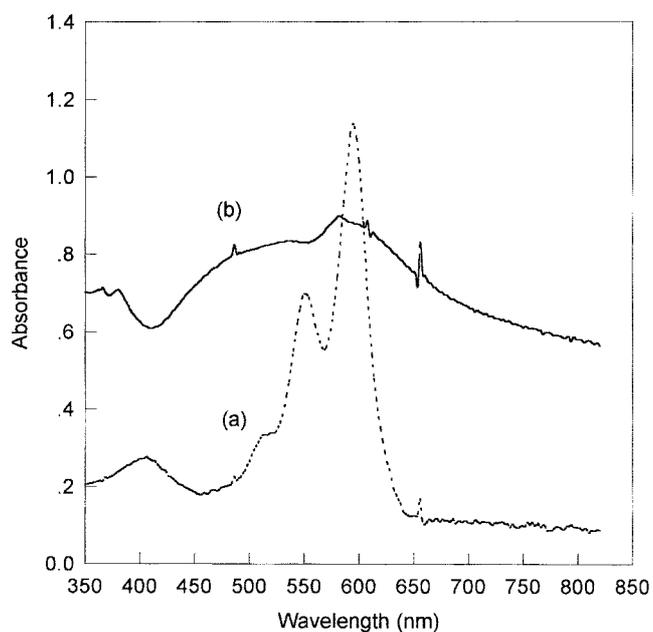


Figure 4. Absorption spectra of perylene 3,4,9,10-tetracarboxyldiimide. (a) $c\text{-H}_2\text{SO}_4$; (b) DMF.

peryene's cation monomer.²⁶ Absorption spectrum in DMF, unlike the spectrum of monomer observed in $c\text{-H}_2\text{SO}_4$, shows absorption characteristics in a broad area. We concluded that this species exists not as a monomer but as a dimer or aggregation.

The characteristics of fluorescence. It is known that research of the fluorescence of a photoconductor is as valuable as a probe of charge generation. And we can determine electron transfer mechanisms of semiconductors adsorbed with dye by observing the luminescence of the semiconductor.³⁰⁻³² Figure 5 shows the fluorescence properties of

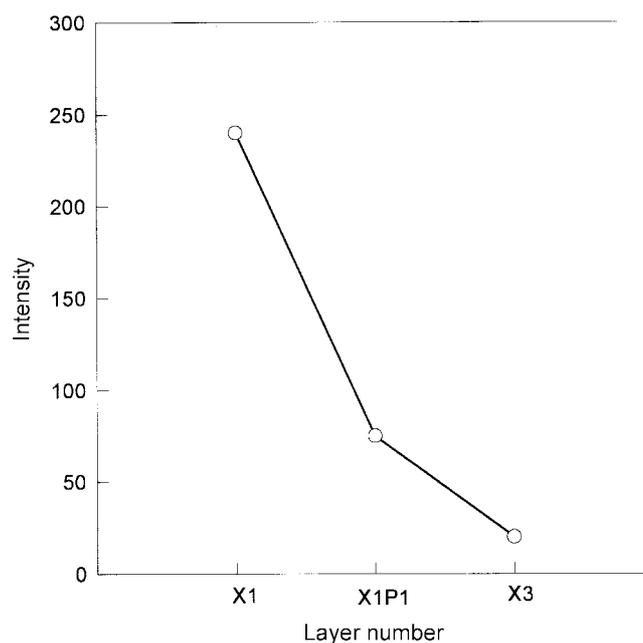


Figure 5. The fluorescence characteristic of X-phthalocyanine.

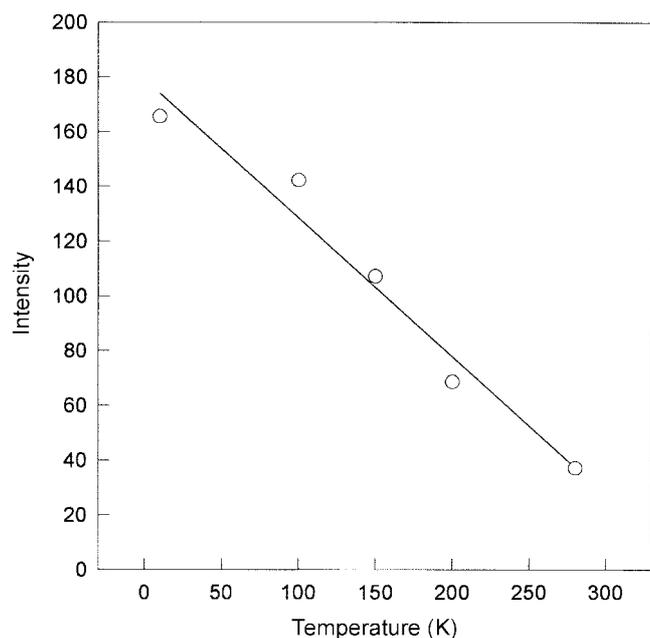


Figure 6. The fluorescence characteristic of perylene 3,4,9,10-tetracarboxydiimide depending on temperature.

phthalocyanine, and as the number of thin film layers increases, the intensity of fluorescence decreases. It is presumed that the fluorescence characteristic of a bilayer electrode formed by X-phthalocyanine and perylene 3,4,9,10-tetracarboxydiimide is inclined to reduction, which means electron transfer occurs between them. Figure 6 shows the fluorescence characteristic of perylene 3,4,9,10-tetracarboxydiimide, and we can assume that electron transfer from perylene 3,4,9,10-tetracarboxydiimide to ITO because the position of the specific peak was not changed and the intensity of fluorescence decreased according to the measurement temperature.

The characteristic of photocurrent. Figure 7 shows the absorption spectra of electrodes made by the LB method and this demonstrates the fact that two compounds exist in the solid state as dimer or aggregation. The absorption characteristics of the thin film used as electrode, X-phthalocyanine keeps on crystal structure, perylene 3,4,9,10-tetracarboxydiimide shift to short wavelength rather than absorption wavelength in the solution state, and the maximum absorption appeared between 450 nm and 600 nm. It is assumed that it is shifted in a short wavelength, means perylene 3,4,9,10-tetracarboxydiimide, like X-phthalocyanine, exists not as a monomer but in the form of a dimer or aggregation. This could be a valuable source for estimating the characteristics of the photocurrent according to the aggregation's crystal structure, and the surface pressure used in the meantime of transfer of thin film does not cause the destruction of the thin film and crystal properties are preserved. Figure 8 shows the photocurrent's characteristics of X-phthalocyanine and perylene 3,4,9,10-tetracarboxydiimide. The characteristics of the photocurrent of electrode made by the LB method were similar to the characteristics of the

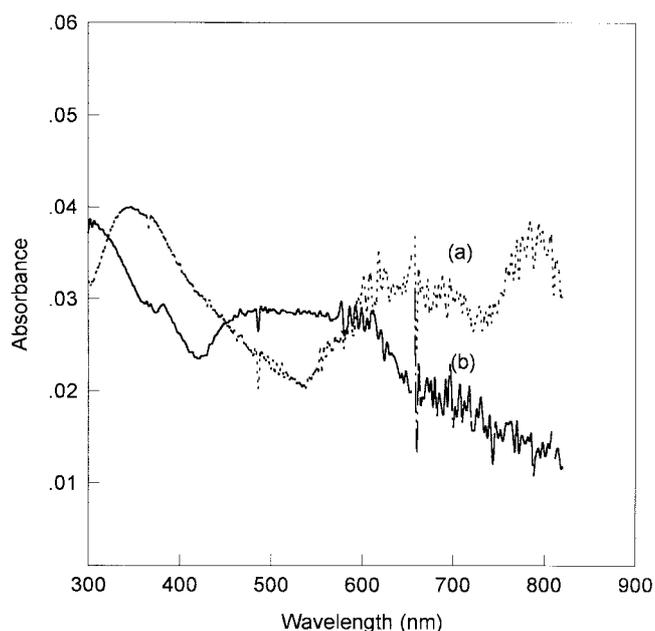


Figure 7. Absorption spectra of thin film prepared on ITO. (a) X-phthalocyanine; (b) perylene 3,4,9,10-tetracarboxydiimide.

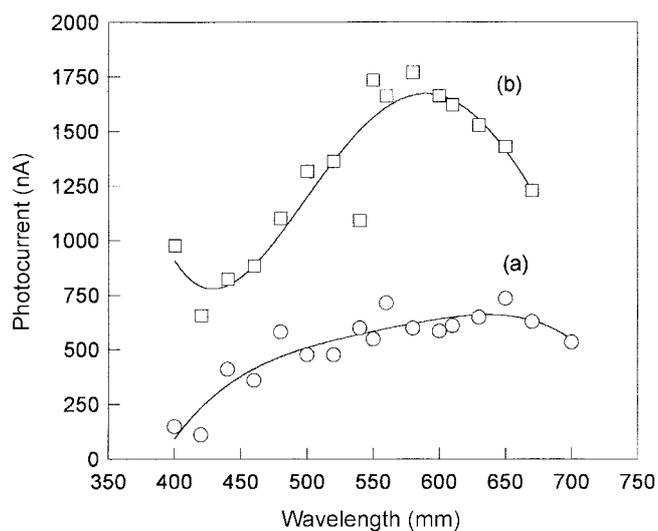


Figure 8. Photocurrent action spectrum of photoreceptor. (no bias). (a) X-phthalocyanine (5 layer); (b) X-phthalocyanine: perylene 3,4,9,10-tetracarboxydiimide (5 : 1 layer).

absorption wavelength in panchromatic wavelength, and it is assumed that the crystal structure produced in the process of electrode preparation influences the photocurrent. In X-phthalocyanine, cathodic photocurrent, the characteristics of p-type semiconductor are observed, and in perylene 3,4,9,10-tetracarboxydiimide anodic photocurrent is observed, which means it has the characteristics of n-type semiconductor. The characteristics of the photocurrent in the case of the applied potential is zero, which is similar to the absorption spectrum of dye in the solid state, and it is observed "in phase" as seen with merocyanine.³³ Figure 9 shows the characteristics of the photocurrent of each electrode. The photocurrent and open photovoltage of X-

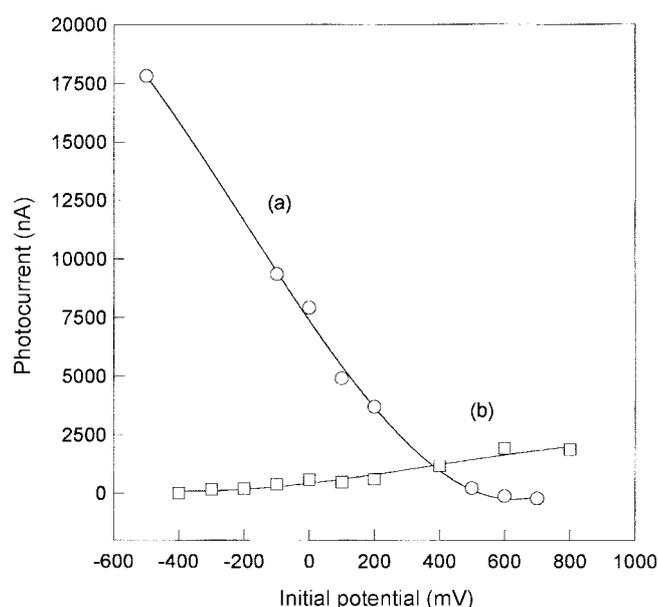


Figure 9. The photocurrent-photovoltage characteristic of photoreceptor. (a) X-phthalocyanine; (b) perylene 3,4,9,10-tetracarboxyldiimide.

phthalocyanine are 7918 nA and 588 mV, respectively, and those of perylene 3,4,9,10-tetracarboxyldiimide are -585 nA and -400 mV, respectively. Positive potential shows photocathodic activity, whereas negative potential shows photoanodic activity. From these values, the possibility of photovoltage devices in the case of X-phthalocyanine seems to be superior. We compared the characteristics of photocurrent by increasing the layers of X-phthalocyanine. As shown in Table 1, as the layers of X-phthalocyanine increase to 3 layer, the photocurrent increases simultaneously. When perylene 3,4,9,10-tetracarboxyldiimide exists in one layer, as the layers of X-phthalocyanine increase, the photocurrent decreases from $2.41 \mu\text{A}$ at X1 to $2.214 \mu\text{A}$ at X3P1. In the case of X5P1, it increases to $35.98 \mu\text{A}$. A hole and electrons of X-phthalocyanine excited by light have no impact on the photocurrent because they recombined with perylene 3,4,9,10-tetracarboxyldiimide on the interfaces, but in the case of X5P1, there exist enough carriers to overcome the reunited coulomb force to increase the photocurrent. To

Table 1. Concentration dependence of phthalocyanine on photocurrent (μA)

Materials	X1	X3	X5	X1P1	X1P3	X3P1	X5P1
1M NaNO ₃	2.41	2.48	2.47	1.09	0.99	2.214	35.98

*X: X-phthalocyanine; P: perylene 3,4,9,10-tetracarboxyldiimide; Layer number: 1=1 layer, 3=3 layer, 5=5 layer.

Table 2. Wavelength dependence of phthalocyanine on photocurrent (nA)

Materials	NaNO ₃		Hydroquinone		Hydrazone		Methylviologene	
	550 nm	620 nm	550 nm	620 nm	550 nm	620 nm	550 nm	620 nm
X1	60.2	39	-9.4	-2.9	0.34	0.31	15	22
X1P1	55.4	52	-10.5	-4.51	0.59	0.862	24	31

investigate the mechanism of photosensitization, we studied the role of additives. We used 4-dibenzylamino-2-methylbenzaldehyde diphenylhydrazone as an electron donor and methylviologen as an electron acceptor. As shown in Table 2, the role of additives was attained as a result of decreasing photocurrent, and such an effect is assumed to be a result of the recombination of the electron-hole pair creating after excitation. It is proved that in the 4-dibenzylamino-2-methylbenzaldehyde diphenylhydrazone charge that was created has not effect on the route of a current stream, but effects the recombination response of electron-hole pair that have been created. The reduced photocurrent of the 4-dibenzylamino-2-methylbenzaldehyde diphenylhydrazone, as we assumed from the fluorescence characteristic, electron transfer, occurs with perylene 3,4,9,10-tetracarboxyldiimide and so the response of recombining occurs first.³⁴⁻³⁵ The role of 4-dibenzylamino-2-methylbenzaldehyde diphenylhydrazone is assumed to reduce anodic photosensitization or cathodic photosensitization.

The characteristics of photoinduced discharge. In the process of photoinduced discharge by light, an electron-hole pair was created at the charge generation layer and a photogenerated hole was moved into the charge transfer layer and neutralized with negative charge on the surface, which explains the mechanism of photogeneration and assumed the process of electron transfer.³⁶ In a dark room after charging the surface of photoreceptor by corona discharge and then creating carrier by the light, and removing surface charge, we can attain a time dependence curve of surface potential. From such curves, the response about the incident light of the photoreceptor is interpreted. Figure 10 shows that when the ratio of the weight percent of X-phthalocyanine and perylene-3,4,9,10-tetracarboxyldiimide is 1 to 1, the photoresponse appears. Surface potential was reduced

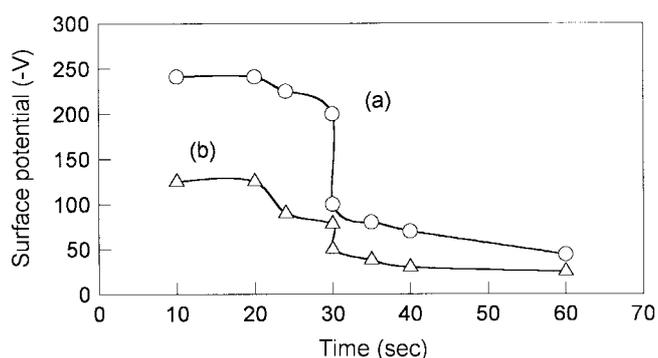


Figure 10. Photoinduced discharge curve of X-phthalocyanine photoreceptor depending on weight percent of perylene 3,4,9,10-tetracarboxyldiimide. (a) 0%; (b) 50%.

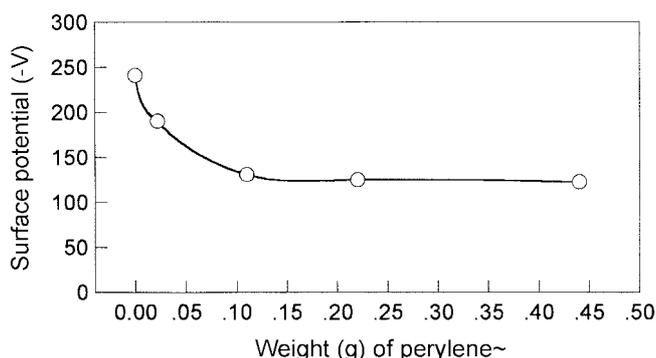


Figure 11. Surface potential effect by addition amount of perylene 3,4,9,10-tetracarboxydiimide for X-phthalocyanine photo-receptor.

by 50% and the speed per second of dark decay was increased about 6V. Also the photoinduced discharge characteristic decreased by 5 times on the basis of the fact that the initial potential was reduced by half. Surface potential can indicate the degree of charge capacity ability, and if it is reduced it is assumed to be lost without preserving charge. It is also presumed that the influence of dark decay decreases due to electron transfer among molecules. Figure 11 shows that the surface potential was changed by the addition of perylene 3,4,9,10-tetracarboxydiimide. The change in surface potential does not appear significant when the amount of addition is above 50%. The characteristics of photoinduced discharge for each weight percent are shown in Table 3. The characteristics of photoinduced discharge

Table 3. Electrophotographic values of X-phthalocyanine

Weight percent of perylene 3,4,9,10-tetracarboxydiimide	0%	33%	50%	67%
V_i (-V)	241	131	125	122.4
dark decay ($\Delta V/\Delta s$)	-41	-44.6	-47	-58.4
$E_{0.5}^*$	0.1	0.8	0.5	1.0

* $E_{0.5}$ is the product of I and t , where t is the time for I to photinduced discharge from V_i to $0.5V_i$ (I : intensity of light).

decreased as the weight percent of perylene 3,4,9,10-tetracarboxydiimide increased, and it is presumed that, as the electron-hole pair that was created by the light is dissociated, the response of recombination occurs before the phenomenon of 4-dibenzylamino-2-methylbenzaldehyde diphenylhydrazone which carries the hole.

Photogeneration Mechanism. The mechanism of photogeneration for a photoconductor is defined as an intrinsic or extrinsic process.³⁶ The conceptual scheme is given in Figure 12. Essentially, after excitation of the photoconductor, the excited photoconductor, in most cases the exciton, can either fluoresce or decay nonradiatively to the ground state. If this excited state is ionized, and regardless of activation mode (heat or electric field assisted), an electron-hole pair is created, and the process of photogeneration is defined as an intrinsic. On the other hand, if the excited state needs to react with an electron donor or electron acceptor before the formation of the electron-hole pair, the photogeneration process is defined as extrinsic. In extrinsic photogeneration, an intermediate bound electron-hole pair, such as an exci-

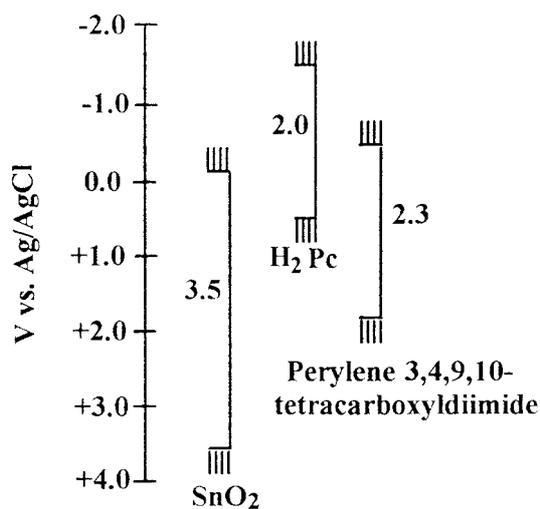


Figure 13. Energy level representation of ITO/phthalocyanine/peryrene 3,4,9,10-tetracarboxydiimide/Pt cell under short circuit condition.

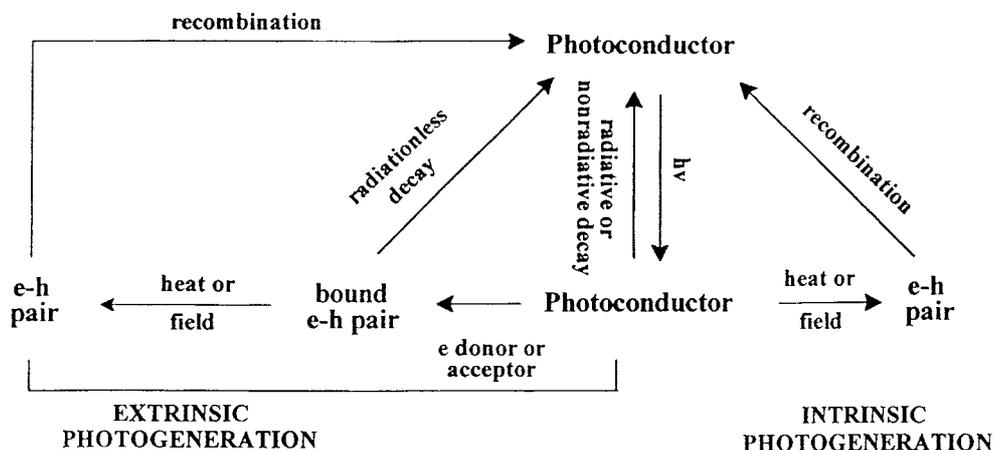


Figure 12. Definitions of the intrinsic and extrinsic photogeneration processes.

plex or excited charge transfer complex, may be involved. A good relationship between the quenching of fluorescence and photogeneration data is a singlet state (S_1) that was initially excited is in the form of precursor for photogeneration. It is presumed that the mechanism of creating a photocurrent from the perspective of energy proceeds as shown in Figure 13. If the perylene 3,4,9,10-tetracarboxyldiimide does exist, in the photogeneration mechanism of X-phthalocyanine, excited X-phthalocyanine forms an exciton and this exciton spreads onto the surface and then forms exciplex with perylene 3,4,9,10-tetracarboxyldiimide. It is presumed that the electron of formed exciplex is delivered into perylene 3,4,9,10-tetracarboxyldiimide and proceeds as a response, being injected as an electrolyte.

Conclusion

The characteristics of photochemistry and photoelectrochemistry have been investigated by introducing perylene 3,4,9,10-tetracarboxyldiimide to expand the region of absorption wavelength as a shorter wavelength (400-600 nm) region than X-phthalocyanine. We have reached the following conclusions. The absorption characteristic of X-phthalocyanine and perylene 3,4,9,10-tetracarboxyldiimide in the solid state exists as a dimer or aggregation rather than a monomer. The fluorescence characteristic of perylene 3,4,9,10-tetracarboxyldiimide decreases as the temperature increases. The maximum photocurrent appears in the same region of absorption wavelength because of the influence of the aggregation structure. In the case of the addition of perylene 3,4,9,10-tetracarboxyldiimide into X-phthalocyanine, the intensity of fluorescence decreases by 69% and the photocurrent decreases by 55%. (at X1PI electrode) Additives is cause hydroquinone to reverse the direction of the photocurrent, and methylviologen and 4-dibenzylamino-2-methylbenzaldehyde diphenylhydrazone proceeded to recombine in the form of a exciplex rather than contributing to the photocurrent, by blocking the photocurrent. The characteristics of photoinduced discharge decreased by a factor of 5 and the speed of dark decay increased by a factor of 1.2.

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