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Effect of pH on Photopolymerization Reaction of Aniline Derivatives with the Tris(2,2′-bipyridyl)ruthenium Complex and the Methylviologen System

Kenjiro Teshima,[†] Sei Uemura,[†] Norihisa Kobayashi,*[,‡] and Ryo Hirohashi[‡]

Graduate School of Science and Technology and Department of Information and Image Sciences, Chiba University, 1-33 Yayoi-cho, Inage-ku, Chiba 263-8522, Japan

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ABSTRACT: Photopolymerization of aniline derivatives was revealed to be induced by photoinduced electron transfer (PIET) between the tris(2,2′-bipyridyl)ruthenium complex [$Ru(bpy)_3^{2+}$] and electroactive molecules. Since the initial photopolymerization reaction depended on PIET between $Ru(bpy)_3^{2+}$ and *N*-phenyl-*p*-phenylenediamine (PPD), the effect of pH of the polymerization solution on the PIET was investigated to improve the efficiency of the photopolymerization reaction. At lower pH, the quenching reaction of $[Ru(bpy)_3^{2+}]^*$ by PPD proceeded with low efficiency due to electrostatic repulsion between molecules concerning the PIET. This was clarified by comparison with the results concerning the photochemistry in a higher pH solution. On the other hand, a lower pH should be required to induce the propagation because a lower pH inhibited the deprotonation of both aniline derivative and growing poly(aniline) chain. From the detailed analyses for both initial and propagation reactions, the increase in ionic strength was revealed to be a solution to improve the photopolymerization efficiency even in low pH.

Introduction

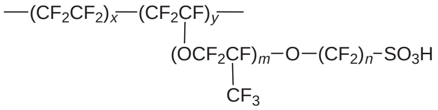
Photoinduced electron transfer (PIET) between $Ru(bpy)_3^{2+}$ and other electroactive molecules has been focused from the standpoint of photosynthesis, photolysis of water, a photoenergy conversion system, and so on.[1] For these applications, inorganic semiconductor particles and deposits give higher efficiency.[2] However, the PIET between molecules has an advantage for easier fabrication of an attractive molecular function system. The fact that $Ru(bpy)_3^{2+}$ is sensitized in the visual wavelength range would be another advantage for the conversion of sunlight compared with an inorganic semiconductor. In this molecular system, the relative location of the redox potential level for the molecules concerning PIET is important for successful PIET. Since varied molecular designs are available in organic molecules and their potential level can be easily estimated with recently developed computer simulations, several approaches with the molecular system for the above application have been reported[3] and are expected to open up a wide range of new applications for molecular devices.

Conducting polymers are also collecting keen interest for the application of some electronic devices. If these conducting polymers are prepared by PIET, this will give new attractive applications for molecular electronics as well as methodologies to analyze the initial polymerization mechanism. On the basis of these interests, photopolymerization of pyrrole has already been performed by using $Ru(bpy)_3^{2+}$ [4] and $Cu(dpp)_2^{+}$,[5] succeeding in the preparation of poly(pyrrole). We also have reported a photopolymerization of an aniline derivative by illuminating the bilayer film composed of a $Ru(bpy)_3^{2+}$-incorporated Nafion film and a methylviologen pendant poly(siloxane) film in an aqueous solution containing PPD and/or aniline.[6] It became clear that PPD, the head-to-tail dimer of aniline, should be required as an initiator to induce the photopolymerization of aniline with PIET of $Ru(bpy)_3^{2+}$. However, the efficiency for the photopolymerization was insufficient. Detailed mechanisms of the PIET reaction and polymerization process still remained to be clarified in order to improve its efficiency. The electrochemical polymerization reaction of aniline involves protonation and deprotonation processes for aniline monomer and growing intermediates. The pH of the polymerization solution should therefore affect the present photopolymerization of aniline derivatives, and the analysis would give the researcher a clue to produce a more efficient system.

In the present paper, we discussed the effect of pH of the polymerization solution on the quenching reaction of $[Ru(bpy)_3^{2+}]^*$ by PPD. The quenching reaction should be the initial reaction for the photopolymerization. On this basis, a improvement in the initial reaction was carried out. Consequently, a more efficient photopolymerization was performed in low pH solution.

Experimental Section

$Ru(bpy)_3^{2+}$ was prepared according to the literature procedure[7] and then purified by recrystallization with water. Reagent grade methylviologen (MV^{2+}) (Tokyo Chemical Industry Co., Ltd.) and HCl (Kanto Chemical Co., Inc.) were used as received. Flemion (**I**) ethanol solution (9 wt %) was given from Asahi Glass Co., Ltd.



$$-(CF_2CF_2)_x-(CF_2\underset{\underset{\underset{CF_3}{|}}{(OCF_2CF)_m-O-(CF_2)_n-SO_3H}}{\overset{}{CF}})_y-$$

An alcoholic solution containing $Ru(bpy)_3^{2+}$ (0.2 wt %), MV^{2+} (0.08 wt %), and Flemion (2.5 wt %) was spread on a glass

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* To whom correspondence should be addressed. Telephone: +81-43-290-3458. Fax: +81-43-290-3490. E-mail: norichan@image.tp.chiba-u.ac.jp.

[†] Graduate School of Science and Technology.

[‡] Department of Information and Image Sciences.

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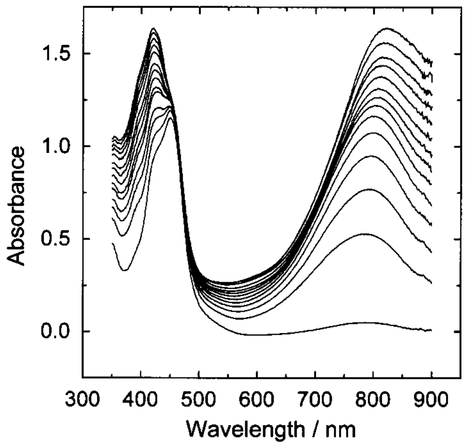


Figure 1. Change in absorption spectrum of a $Ru(bpy)_3^{2+}$ + MV^{2+} + Flemion single layer film illuminated in a 2.0 M HCl aqueous solution containing 0.3 M aniline and 1.0 mM PPD.

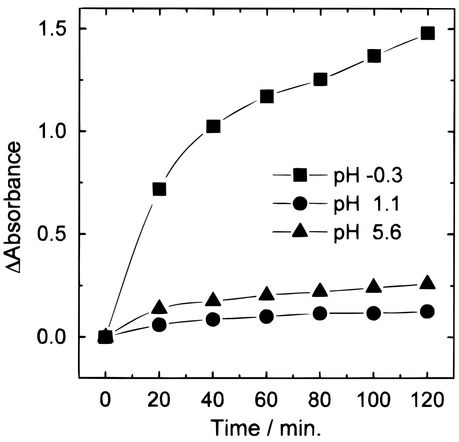


Figure 2. Changes in absorbance at 800 nm of $Ru(bpy)_3^{2+}$ + MV^{2+} + Flemion single layer films in the polymerization solution upon continuous illumination.

substrate, and the resulting $Ru(bpy)_3^{2+}$ + MV^{2+} + Flemion single layer film was dried at 353 K for 8 h *in vacuo*. An HCl aqueous solution containing aniline (0.3 M) and PPD (1.0 mM) was used as the polymerization solution. The single layer film was immersed in the polymerization solution and was illuminated in the visible region. ($\lambda = ca.$ 420−600 nm, 89.5 mW cm^{-2}) from the glass substrate side with a 500 W xenon lamp (Ushio Inc.).

The photoemission intensity of $Ru(bpy)_3^{2+}$ (1×10^{-5} M) was measured with fluorescence photospectrometer F-4500 (Hitachi Co., Ltd.) in an HCl aqueous solution containing PPD and aniline (0.3 M) under a nitrogen atmosphere. The excitation of $Ru(bpy)_3^{2+}$ was performed at 450 nm, and the photoemission intensity was monitored at 600 nm.

Result and Discussion

The single layer film containing both $Ru(bpy)_3^{2+}$ and MV^{2+} was employed in the present experiment unlike in our earlier report[6] in order to improve the photopolymerization of aniline derivatives for the film. Since the polymer is also produced in the polymerization solution during illumination, it is favorable from the viewpoint of efficient image formation of the conducting polymer to reduce the amount of the poly(aniline) produced in the solution. Figure 1 shows the change in the absorption spectrum for the film alone upon illumination. In this figure, the contribution from the absorption of the poly(aniline) that existed in the polymerization solution was neglected in order to estimate the amount of photopolymerized poly(aniline) for the film alone. The absorption peaks at around 800 and 400 nm are assigned to the conjugated polaron structure and the semiquinone cation radical structure of poly(aniline).[8,9] The enhancement of the absorbance at 800 nm indicates that the photopolymerization of the aniline derivative is induced for the single layer film under continuous illumination conditions. The enhancement of the absorption was comparable to the previously reported one,[6] which included a contribution from the absorption of poly(aniline) in the polymerization solution. The previous study was carried out with a bilayer system composed of $Ru(bpy)_3^{2+}$ containing Nafion film and a MV^{2+} pendant poly(siloxane) film, and more than half the amount of poly(aniline) was produced in the polymerization solution. It is difficult to compare this result with the previous one quantitatively because the thickness of the film and the concentration of MV^{2+} are different (the concentration of $Ru(bpy)_3^{2+}$ is the same). However, by comparing the change in the absorption upon illumination, the single layer structure seems to be effective to produce photopolymerized poly(aniline) for the film. This is probably attributed to an increase in the probability that $Ru(bpy)_3^{2+}$ and MV^{2+} come into contact or very nearly so in the single layer film, because $Ru(bpy)_3^{2+}$ and MV^{2+} are homogeneously dispersed in the film. Electron transfer between $Ru(bpy)_3^{2+}$ and MV^{2+} can take place at any place in the single layer film unlike that at the only interface between $Ru(bpy)_3^{2+}$ and MV^{2+} films in the bilayer system. The following studies were carried out with single layer film.

As described in the Introduction, the pH of the aniline polymerization solution considerably influences its electropolymerization because polymerization of aniline involves protonation and proton release for the growing poly(aniline) chain. If such a polymerization mechanism is applied to the other aniline polymerization, the present photopolymerization should also be affected considerably by the pH of the solution, and it is of interest and useful for effective image formation of the conducting polymer to analyze the effect of pH on the photopolymerization of the aniline derivative for the film. As a result of this inference, the single layer film was immersed in three distinct pH polymerization solutions (pH = 5.9, 1.1, −0.3), and the three solutions were illuminated from a xenon lump through a filter. Figure 2 shows the change in the absorbance at 800 nm for the film alone upon illumination in a given pH solution. The photopolymerization leading to poly(aniline) formation for the film was induced effectively by employing the solution at pH −0.3. On the other hand, for the solutions at pH 5.9 and even pH 1.1, where conductive poly(aniline) can be commonly obtained by electropolymerization, the formation of poly(aniline) for the film was not observable to the naked eye while the absorbance at 800 nm slightly increases upon illumination. This result indicates that the photopolymerization leading to poly(aniline) for the film is considerably affected by pH of the solution but its mechanism seems to be different from common electropolymerization. In order to estimate the structure of products on the film prepared at each pH, each absorption spectrum was compared and analyzed. The difference absorption spectrum for the film after the illumination of single layer film for 2 h is shown in Figure 3. The larger absorption change for the film prepared at pH −0.3 indicates that the photopolymerization is induced at a lower pH. At pH 1.1, photopolymerized poly(aniline) is

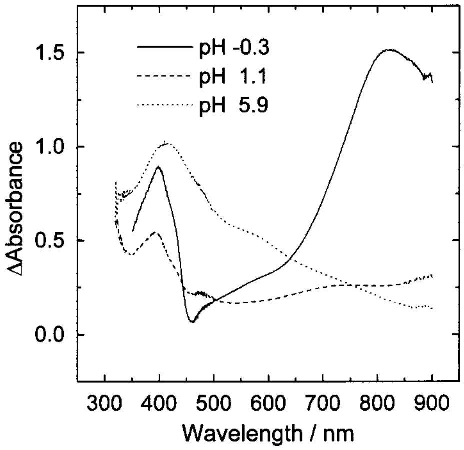


Figure 3. Difference spectra of single layer film after 2 h of visible illumination in each pH polymerization solution.

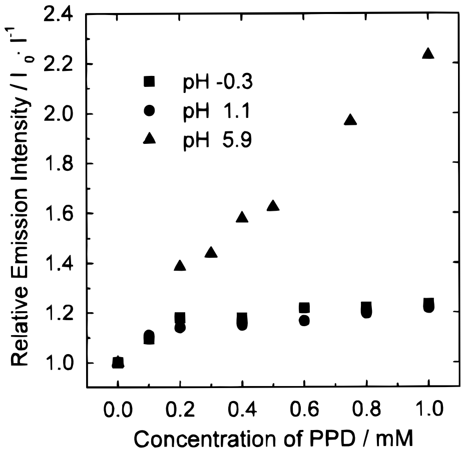
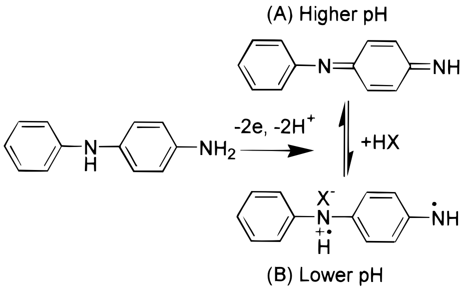


Figure 4. Stern−Volmer plots for the quenching reaction of the emission of [Ru(bpy)$_3$$^{2+}$]* by PPD in each HCl aqueous solution, containing 0.3 M aniline.

Scheme 1. Electrochemical Reaction of PPD in Each pH Solution



apparently prepared for the film as is seen from the result that the absorbances at both 400 and 800 nm are slightly increased. On the other hand, at pH 5.9, no enhancement of the absorbance at 800 nm was observed, but absorption peaks at 410 and 589 nm appeared. It has already been reported that these absorption are found for oligomers ($n \approx 4$) of aniline derivatives.[10] This suggests that illumination for the single layer film in the solution at pH 5.9 does not lead to poly(aniline) but to oligomers of aniline with lower molar masses.

Polymerization of aniline is well-known to be initialized by generating cation radicals by oxidation of aniline.[11] In the present photopolymerization, cation radicals should be generated via photooxidation of PPD by Ru(bpy)$_3$$^{2+}$. In order to analyze the effect of pH on initial reaction for the photopolymerization in detail, the quenching reaction of [Ru(bpy)$_3$$^{2+}$]* by PPD was evaluated by comparing the emission intensity of [Ru(bpy)$_3$$^{2+}$]* in solution at each pH as a function of PPD concentration. Its Stern−Volmer plot is shown in Figure 4. The relation for pH 5.9 was linear, but those for pH 1.1 and −0.3 deviated from the linearity expected for the dynamic electron transfer process, except at low PPD concentration. This indicates that the quenching reaction of [Ru(bpy)$_3$$^{2+}$]* by PPD is inhibited with increasing PPD concentration. A possible explanation of this difference would lie in the effect of pH on the electronic state of PPD. Namely, PPD should be fully positively charged at a pH lower than −0.1 due to protonation to both amino and imino groups but should be neutral at pH 5.9. Electrostatic repulsion between positively charged Ru(bpy)$_3$$^{2+}$ and PPD may affect the electron transfer process between these molecules. Detailed discussion on this tendency will be given later.

In order to compare the rate constant of the electron transfer between Ru(bpy)$_3$$^{2+}$ and PPD at each pH, a linear line was drawn through the plots at lower PPD concentrations, particularly for pH 1.1 and −0.3, because those plots showed a linear relationship at low PPD concentrations. This estimation seems to be less correct for the total kinetics but makes its interpretation simple. The estimated quenching rate constants for pH 5.9, 1.1, and −0.3 were 2.6×10^9, 1.5×10^9, and 2.0×10^9 M^{-1}·s^{-1}, respectively. Each quenching rate constant is almost similar to but is higher than that between Ru(bpy)$_3$$^{2+}$ and MV^{2+} (4.5×10^8 M^{-1}·s^{-1}).[12] This suggests that efficient reductive quenching reaction occurs between [Ru(bpy)$_3$$^{2+}$]* and PPD.

Contrary to the fact that the photopolymerization was induced in the solution at pH −0.3 but not at pH 1.1 and 5.9, the quenching reaction at pH −0.3 was strongly restricted at higher PPD concentrations. Taking into account that the present quenching reaction was due to PIET from PPD to [Ru(bpy)$_3$$^{2+}$]*, this result indicated that the PIET should be a key factor as an initial reaction to enhance photopolymerization, but it did not work effectively in the solution at lower pH. The reason why photopolymerization was not induced at higher pH (i.e., pH 5.9) was due to deprotonation from the growing chain. For the electropolymerization of aniline in the solution at higher pH, it is well-known that deprotonation from the growing poly(aniline) chain occurs and the resulting polymer becomes electrochemically inactive. Such a kind of deprotonation should also take place for aniline, PPD, and the growing poly(aniline) chain in the course of photopolymerization. Coupling and propagation reactions for PPD and/or oligomers should be required to obtain a polymer with higher molar mass even in the case of photopolymerization. Since the quinone diimine form does not show coupling activity because of its stability, the growing intermediate should have a protonated form, the anilinium radical form, as shown in Scheme 1. Namely, the pH of the photopolymerization solution plays an important role in the coupling and propagation reactions. In the present experiment, the growing intermediate would have the quinone diimine form at pH 5.9. Therefore, coupling and propagation reactions were restricted at the oligomer level.

In order to analyze the details of pH effect on, especially, the initial reaction and to improve the initial reaction at lower pH, the emission intensity of Ru(bpy)$_3$$^{2+}$ in the HCl aqueous solution containing 1.0 mM

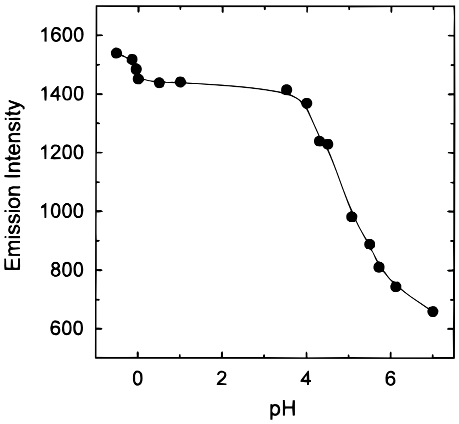


Figure 5. Dependence of the pH of an HCl solution containing 0.3 M aniline and 1.0 mM PPD on the emission intensity of $[Ru(bpy)_3^{2+}]^*$.

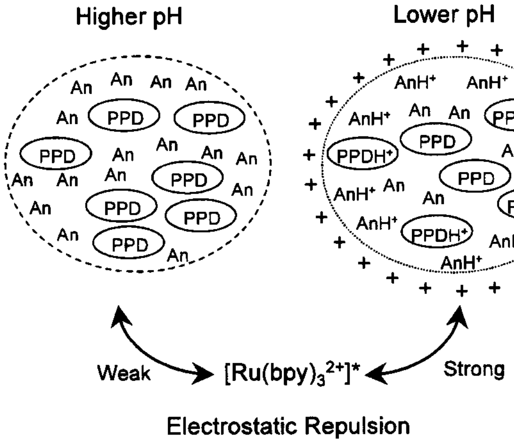


Figure 6. Schematic diagram for electrostatic repulsion between $[Ru(bpy)_3^{2+}]^*$ and the PPD particle in each solution.

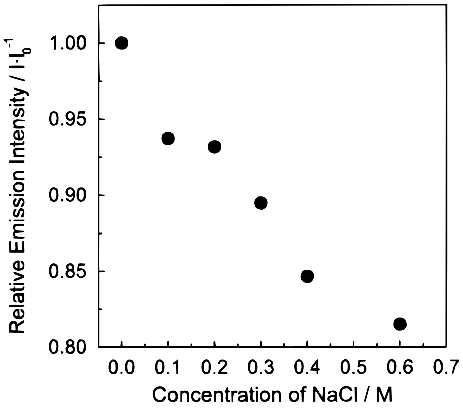


Figure 7. Dependence of the NaCl concentration on the emission intensity of $[Ru(bpy)_3^{2+}]^*$ in an HCl aqueous solution containing 1.0 mM PPD and 0.3 M aniline.

PPD and 0.3 M aniline was plotted as a function of pH as shown in Figure 5. The emission intensity was decreased steeply at around pH 4 with increasing pH. As mentioned above, aniline and PPD have the amino or imino structure. Equilibrium between the protonated form and the deprotonated form should be established depending on pH, since the acid dissociation constants of PPD and aniline are known to be 5.6 and 4.6,[13] respectively. It is noted that PPD is fully protonated (positively charged) at pH values lower than −0.1; the aspect for the quenching reaction as shown in Figure 5 is probably concerned with the equilibrium for aniline. In order to summarize the above-mentioned phenomena, we schematically illustrated the form and situation of PPD and aniline in the polymerization solution as shown in Figure 6. This representation was based on the experimental fact that PPD showed poor solubility in an HCl aqueous solution as the polymerization solution, i.e., 1 mM of PPD could not be solubilized in 2 M HCl aqueous solution unless 200 W sonication was performed for over 30 min. Namely, for the preparation of the polymerization solution in the present experiment, PPD was first dissolved in aniline, and then the resulting solution was mixed with HCl aqueous solution in order to prepare the homogeneous polymerization solution instantly. When the resulting solution containing PPD and aniline was allowed to stand for 1 day, precipitates of PPD were found in the solution. From this experimental fact, we presume that PPD would be surrounded by anilines to form a PPD−aniline particle due to hydrophobic interaction as described in Figure 6, while the solution appeared homogeneous to the naked eye. On this basis, we can rediscuss the difference in the Stern−Volmer plots at each pH, as shown in Figure 4, in terms of electrostatic repulsion between $Ru(bpy)_3^{2+}$ and a PPD−aniline particle. At pH lower than 4.6, both aniline and PPD should be protonated. Therefore, the surface on the PPD−aniline particle should be multiply positively charged. This probably prevents the diffusion of positively charged $Ru(bpy)_3^{2+}$ near the surface of the PPD−aniline particle due to electrostatic repulsion decreasing the rate of electron transfer between $[Ru(bpy)_3^{2+}]^*$ and PPD. With increasing PPD concentration, the possibility for incorporation of PPD molecules in the core of the PPD−aniline particle would be increased. Since such an incorporated PPD cannot easily participate in the PIET due to the great distance between $Ru(bpy)_3^{2+}$ and the incorporated PPD, this results in a deviation from the linear Stern−Volmer relationship, as shown in the plots for pH 1.1 and −0.3. On the other hand, at pH higher than 4.6, aniline should be in the deprotonated form. Since the electrostatic repulsion is reduced due to the less positive surface of the PPD−aniline particle, positively charged $[Ru(bpy)_3^{2+}]^*$ can diffuse near its surface. This results in an easier quenching reaction of $[Ru(bpy)_3^{2+}]^*$ by PPD at pH 5.6 in Figure 4. Even at pH over 5.6, some of the PPD molecules are incorporated in the core of the PPD−aniline particle at higher PPD concentrations and they may exist in the core far from the surface of the particle. Since $Ru(bpy)_3^{2+}$ can be easily dissolved in deprotonated aniline, the diffusion of $Ru(bpy)_3^{2+}$ into the PPD−aniline particle may contribute to the linearity of the Stern−Volmer plots at higher PPD concentrations at pH 5.6 in Figure 4.

The above results remind us that the decrease in electrostatic repulsion between $Ru(bpy)_3^{2+}$ and the PPD−aniline particle leads to efficient photopolymerization. Generally, it is known that the electrostatic repulsion between molecules in the solution can be reduced by an increase in the ionic strength. The quenching reaction of $[Ru(bpy)_3^{2+}]^*$ by PPD was therefore analyzed in the solution in the presence of NaCl in order to justify our claims and to prepare a higher efficient photopolymerization system. The dependence of NaCl concentration on the emission intensity of $Ru(bpy)_3^{2+}$ in an HCl aqueous solution containing PPD (1 mM) and 0.3 M aniline at pH −0.3 was shown in Figure 7. The emission intensity decreased with NaCl concen-

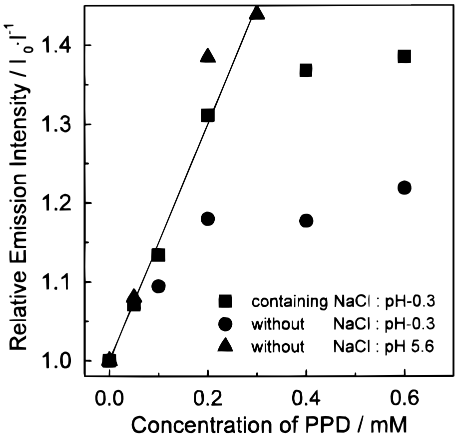


Figure 8. Stern−Volmer plots for the quenching reaction of the emission of $[Ru(bpy)_3{}^{2+}]^*$ by PPD in each HCl aqueous solution, containing 0.3 M aniline, in the presence or absence of 0.4 M NaCl.

tration, suggesting that the quenching reaction was facilitated by increasing the ionic strength. This seems to support our claims. However, the shift of the equilibrium from a protonated PPD to an unprotonated one by increasing the ionic strength may be considered as the alternative explanation for this phenomena, because protonated PPD would be a poorer electron donor. Namely, increasing the ionic strength shifts the equilibrium in favor of the unprotonated PPD, thereby increasing the PIET. In order to evaluate the validity of this explanation, UV−vis absorption spectra and cyclic voltammograms (CV) were measured for the solution in the presence and absence of NaCl. From the result of the absorption spectra, there was no difference between the solution in the presence and absence of NaCl. Since protonated PPD has a different spectrum from unprotonated PPD, this suggests that the equilibrium does not shift by addition of NaCl. In addition, we investigated the oxidation potential in CV for the solution in the presence and absence of NaCl to analyze the equilibrium shift. Since no difference was found between CVs, this supports the point that an equilibrium shift does not occur. These results support that the electrostatic repulsion is unique explanation for the enhancement of the PIET between $[Ru(bpy)_3{}^{2+}]^*$ and PPD in ionic media. Further, when Stern−Volmer plots for the quenching reaction of $Ru(bpy)_3{}^{2+}$ by PPD were evaluated in the presence of 0.4 M NaCl as shown in Figure 8, the plots remained linear at higher PPD concentrations than was true for the case in the absence of NaCl. In addition, these relative emission intensities in the presence of NaCl had higher values at each PPD concentration. Therefore, the difference in the quenching behavior for each pH is believed to reflect an effect of electrostatic repulsion between $Ru(bpy)_3{}^{2+}$ and the PPD−aniline particle. The decrease of electrostatic repulsion makes the quenching reaction easy, because $Ru(bpy)_3{}^{2+}$ can diffuse into the PPD−aniline particle to interact with inner PPD molecules. On the basis of these results, photopolymerization of aniline derivatives was carried out in a solution containing 0.4 M NaCl, and the time course of absorbance changes at 800 nm by continuous illumination for the HCl aqueous solution containing 0.3 M aniline, 1.0 mM PPD and 1.0×10^{-5} M $Ru(bpy)_3{}^{2+}$ in the absence or presence of 0.4 M NaCl was shown in Figure 9. Since this photopolymerization was evaluated in homogeneous solution system, the increase in the absorbance at 800 nm even in the absence of NaCl was faster than that for a single layer film system. As can be seen in Figure 9, it is obvious that the efficient photopolymerization is induced in the presence of NaCl and that Δ absorbance of 2.0 can be obtained by continuous illumination for only 3 min. Since this photopolymerization depends on the intensity of the light source, employment of a laser can realize faster photopolymerization, leading to pattern formation. This efficient photopolymerization in the presence of NaCl supports the point that the electrostatic repulsion between $Ru(bpy)_3{}^{2+}$ and the PPD−aniline particle plays an important role in the photopolymerization of aniline derivatives.

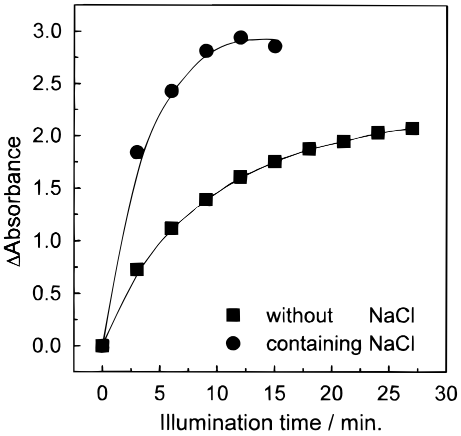


Figure 9. Change in absorbance at 800 nm of an HCl aqueous solution containing 0.3 M aniline, 1.0 mM PPD, and 1.0×10^{-5} M $Ru(bpy)_3{}^{2+}$ in the presence or absence of 0.4 M NaCl upon visible illumination.

Conclusion

The effect of pH on photopolymerization of aniline derivatives with PIET between $Ru(bpy)_3{}^{2+}$ and electroactive molecules was analyzed, and the resulting photoinduced patterning of poly(aniline) was discussed. PIET between $Ru(bpy)_3{}^{2+}$ and PPD was deeply affected by pH of the polymerization solution. The lower pH solution showed less efficient electron transfer, despite the fact that the photopolymerization of the aniline derivative was induced in the solution. From the fact that PPD showed poor solubility in the HCl aqueous solution but good solubility in aniline and the fact that the PIET was restricted below the pH corresponding to acid dissociation of aniline, PPD would be surrounded by anilines to form a PPD−aniline particle due to a hydrophobic interaction. The electrostatic repulsion between $Ru(bpy)_3{}^{2+}$ and the positively charged PPD−aniline particle restricted the PIET as the initial reaction of the photopolymerization. An increase in ionic strength in the photopolymerization solution enabled efficient PIET even in the lower pH solution where the propagation of aniline derivatives proceeded effectively to form poly(aniline). The efficient photopolymerization was therefore achieved by increasing the ionic strength in the lower pH polymerization solution.

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