Electric-Field-Induced Deposition of Rubrene: Preparation of aqueous colloidal suspensions with ionic surfactant and exploration of deposition conditions

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Abstract

Deposition of large-area rubrene film in a cost- and environment-friendly manner without vacuum deposition is essential for the deployment of photon upconversion sheets based on rubrene via triplet-triplet annihilation. In this study, the electric-field-induced deposition of rubrene from aqueous suspensions stabilized by anionic (SDS) or cationic (DTAB) surfactants has been successfully demonstrated. A systematic exploration of the deposition conditions revealed that the voltage required for uniform deposition of colloidal particles over the electrode is much higher than triggering water electrolysis, suggesting that the electric-field-assisted deposition achieved is not simple electrophoretic deposition but is strongly influenced by water electrolysis.

Keywords: electric-field-induced deposition, electrophoretic deposition, rubrene, colloidal suspension

1. Introduction

Electronic devices using organic semiconducting molecules, including polymers, such as organic light-emitting diodes (OLEDs), organic transistors, and organic photovoltaic cells, are promising candidates for key components of next-generation large-area, flexible electronic systems. Large-area deposition of target molecules is an essential technology for fabricating devices with electronic and optical functionality. Wet deposition methods, which do not require strict atmospheric control using large vacuum chambers, have the potential to produce large-area organic electronic devices at reasonable economic and environmental costs. [1-3]

Typical wet deposition methods employ solution materials, which are target molecules dissolved in a solvent. Although spin-coating is widely used for laboratory-level device prototyping because high-quality thin films suitable for device applications are easily obtained through the method, it has low material utilization efficiency because most of the applied material is blown away. In addition, spin-coating is not suitable for patterned deposition or film formation on curved surfaces. Printing methods, such as inkjet printing, address these shortcomings but require countermeasures against difficulties like nozzle clogging and have not yet been deployed for practical use. There seems to be fundamental problems with solution-based deposition methods. These include the need for highly concentrated solutions, the necessity of an acrobatic choice of solvents for multilayer deposition, and the difficulty of separating morphology control of target molecules from the drying process. Some of them stem from the fact that drying the solution and solidifying the target molecules simultaneously take place in solution-based deposition.

Deposition methods employing colloidal suspensions of target molecules may potentially avoid the drawbacks of solution-based deposition methods, because the solidification of target molecules occurs in advance of film formation and drying. One of the typical deposition methods from colloidal suspensions is electrophoretic deposition, in which an electric field drives colloidal particles of target molecules in an insulating dispersion medium to deposit on an electrode. [4,5] We previously reported the fabrication of films by electrophoretic deposition from colloidal suspensions obtained by reprecipitation.[6] The technique enables the deposition of an equal weight mixture of unmodified fullerene C_{60}, which was thought to be poorly soluble in common organic solvents such as toluene, with conjugated polymers including poly(*p*-phenylene vinylene) derivatives and polythiophene derivatives, as well as the fabrication of prototype photovoltaic cells. [7-9] Recently, the possibility of controlling the formation process of C_{60} colloidal particles by blending conjugated polymers has also been demonstrated. [10] The use of non-toxic aqueous dispersion mediums will reduce the environmental cost of device production. [11]

Rubrene is a representative small-molecule semiconductor that has recently attracted attention as a photon upconversion material system, converting near-infrared light to visible light via triplet-triplet annihilation.[12, 13] The system enables power generation by a photovoltaic cell from long-wavelength light that the cell does not directly harvest. [14-17] Furthermore, Izawa *et al.* recently reported the realization of ultralow-voltage driven visible-light OLEDs using the system. [18] The material systems of this class consist of rubrene as the emitter in combination with sensitizer molecules. A well-known strategy to achieve high conversion efficiency is to construct a bilayer with a relatively well-defined boundary rather than a simple mixture. Because it is difficult to fabricate a bilayer with a well-defined boundary using solution-based deposition methods alone, rubrene films have been fabricated by thermal deposition in past studies.

On the other hand, film formation from colloidal suspensions, including electrophoretic deposition, is advantageous for the economical fabrication of large-area bilayers with well-defined boundaries, because it is performed in a poor solvent. There has never been a successful film-forming method for rubrene using a suspension-based approach reported. The original objective of the present study was to find a film-forming method for rubrene using an electric-field-assisted deposition like electrophoretic deposition. Therefore, we initially attempted to fabricate rubrene films by electrophoretic deposition using suspensions with an insulating organic dispersion medium, similar to our preceding studies. However, it had been revealed that the preparation of stable rubrene suspensions was only achieved by using a surfactant aqueous solution as a poor solvent. Here, we report the successful fabrication of rubrene films by electric-field-induced deposition with this type of suspension.

2. Experimental

Figure 1 shows the molecular structures of the main substances used in this study. All reagents, including rubrene (Wako Pure Chemicals, Japan, >90.0%), sodium dodecyl sulfate (SDS, TCI, >85.0%), dodecyltrimethylammonium bromide (DTAB, TCI, Japan,

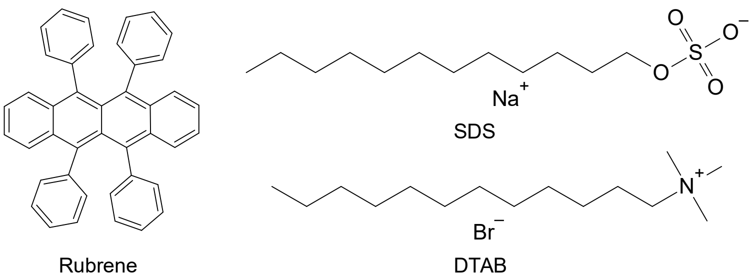

Figure 1 Molecular structures of the main substances used in this study.

>98.0%), and tetrahydrofuran (THF, Wako Pure Chemicals, Japan, >99.5%), were used as received.

To avoid readers rediscovering the same pitfall, we begin by describing how our attempt to prepare a rubrene suspension using an insulating organic dispersion medium failed. In previous studies, suspension preparation using the reprecipitation method was carried out by injecting a toluene solution of the target molecule/polymer into an excess amount of acetonitrile, a poor solvent. [6-10] However, injection of 0.3 ml of a toluene solution of rubrene (10 g/L) into 2.7 ml of acetonitrile, a poor solvent for rubrene, only resulted in a clear solution without colloidal particle formation. Similar results were obtained when the poor solvent was changed to acetone, 1-propanol, 2-propanol, ethanol, and methanol. For the purpose of studying photophysicochemical processes, a successful preparation of rubrene colloidal suspension by injecting a THF solution of rubrene into pure deionized water was reported. [19] However, the suspension prepared in this manner was found to be highly susceptible to sedimentation and not stable enough to be used in electrophoretic deposition.

A stable but dilute rubrene suspension for chemical sensor application was reported to be prepared using aqueous SDS solution as a poor solvent. [20] We modified the procedure to prepare a rubrene suspension suitable for electric-field-induced deposition. Specifically, injecting 0.3 mL of rubrene THF solution (20 g/L) into 2.7 mL of aqueous SDS solution (7.5 mM) under stirring at 800 rpm at 40 °C kept on a hot plate yields a relatively stable colloidal suspension of rubrene with a concentration of 2.0 g/L. Aqueous DTAB solution (13.3 mM) was also found to work as a poor solvent. The concentration of SDS was the same as that reported in Ref. 20, and the value is 92.6% of the critical micelle concentration (cmc) 8.1 mM. The concentration of DTAB was determined to be

92.6% of the cmc 14.1 mM. The cmc values were taken from the literature. [21]

Figure 2 shows a schematic diagram of the setup used for electric field-induced deposition. A U-shaped 3 mm-thick silicone rubber sheet was sandwiched between two electrodes to build a temporary deposition cell. One electrode, an ITO-coated glass, served as a positive electrode. On the other hand, a Ni plate was chosen as a negative electrode instead of an ITO-coated glass plate, because ITO rapidly deteriorates when electrochemically reduced in an aqueous electrolyte [22, 23]. A rubrene colloidal suspension was filled between the two electrodes, and a DC voltage was applied to initiate the deposition.

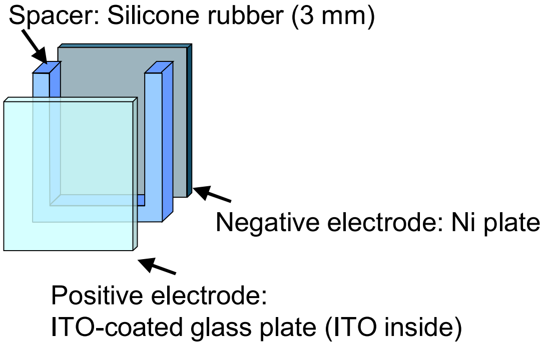

Figure 2 Schematic diagram of the setup used for electric field-induced deposition.

The optical transmission spectra of samples were measured with a spectrophotometer (Shimadzu UV-1800, Japan). Optical glass cells with 1mm in optical path were used for liquid samples. All experiments were performed in air at room temperature.

3. Results and Discussion

Figure 3 (a) shows a photograph of a 2.0 g/L rubrene suspension with aqueous SDS solution as dispersion medium. A light scattering by rubrene colloidal particles is distinctively observed. The suspension obeys a homogeneous red color, characteristic of solid powders, distinct from that of a rubrene solution. Figure 3 (b) shows the log(1/T) spectrum, where T is the optical transmittance, of the suspension. While log(1/T) is a value measured identically to the absorbance of a clear sample, we choose this notation because it has a broad structureless tail over the measured wavelength range originating from light scattering by colloidal particles. The spectrum consists of the tail and optical absorption originating from the electronic transition of rubrene at around 500 nm.

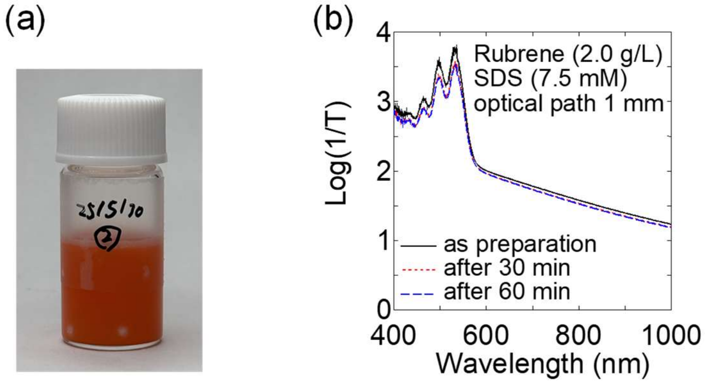

Figure 3 (a) Photograph of a 2.0 g/L rubrene suspension with aqueous SDS solution as dispersion medium (b) Variation of log(1/T) spectrum during standing for 60 min after preparation of the suspension.

Although 1-mm optical glass cells, which have the shortest optical path among those commonly available, were used, the values around these peaks were close to the upper measurement limit of the spectrophotometer, and some noise was observed in the log(1/T) values. However, in this study, the purpose of measuring log(1/T) spectra was to estimate the stability of the suspension as well as the change in the concentration of colloidal particles in the suspension due to voltage application, and a detailed examination of the intensity ratio or wavelengths of the sample peaks is beyond the scope of this study.

The spectrum did not show notable change for 60 min after preparation of the suspension. This confirms that the suspension was relatively stable and did not exhibit any noticeable sedimentation. It has been noticed that the spectral shape as well as the stability of the suspension tends to vary trial by trial.

Figure 4 (a) shows the results of field-induced deposition performed by applying a DC 5 V for 10 min to the rubrene suspension with an SDS aqueous solution as a poor solvent. The deposition of a film consisting of rubrene colloidal particles is clearly observed on the positive ITO electrode. On the other hand, no deposition occurred on the negative Ni electrode. This result indicates that the deposited rubrene colloidal particles have a negative charge. On the other hand, rubrene particles stabilized with DTAB, a cationic surfactant with an alkyl chain length identical to that of SDS, were deposited on the negative Ni electrode as shown in Fig. 4 (b).

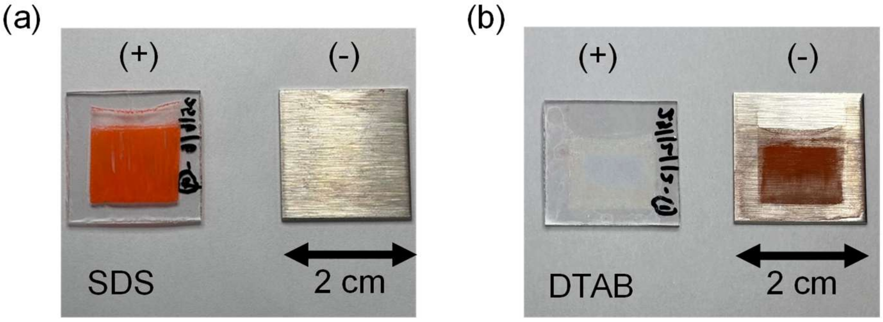

Figure 4 Photographs of electrodes after application of DC 5.0 V for 10 min in the rubrene suspension. (a) and (b) correspond to SDS and DTAB, respectively.

Typically, the stabilization of colloidal particles by ionic surfactants such as SDS and DTAB is provided by the electrostatic repulsion force between the charges of the surfactant ions surrounding the particles. [24,25] Because SDS and DTAB are anionic and cationic, respectively, the observed deposition on the electrode is consistent with the electrophoretic deposition mechanism. However, a relatively high concentration of the surfactant aqueous solution used as the poor solvent provides an ionic concentration in the dispersion medium sufficient to promote an electrochemical reaction on the electrode. In fact, significant gas generation due to water electrolysis was observed during the deposition, which is not observed in electrophoretic deposition using suspensions with an insulating dispersion medium. This indicates that water electrolysis plays a significant role in addition to the electrophoretic process in the electric-field-induced deposition of rubrene observed in this study. This finding suggests that further detailed investigation of the deposition mechanism is required to clarify how the coupling of electrophoretic motion and electrolysis uniquely drives the deposition of rubrene, potentially revealing a new class of electrochemically assisted assembly processes. [26]

The film shown in Fig. 4(a) appears so thick that measuring its optical absorption spectrum using an ordinary spectrophotometer is not possible. Thus, the next target is to obtain a reasonably thin film that can be characterized by an ordinary spectrophotometer. It is reasonable to expect that the reduction of the amount of rubrene deposited may result in a thinner rubrene film. Therefore, the following two experiments were conducted: one in which the application time was reduced while the applied voltage remained at 5 V, and the other in which the voltage was varied from 1 to 5 V while the application time remained 10 minutes.

The results of the reduction of the voltage application time are shown in Fig. 5. It was found that film deposition did not progress gradually over the entire electrode in contact with the suspension, but partial deposition occurred on the edges, especially the

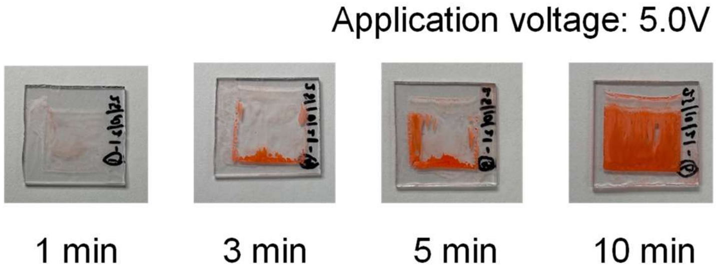

Figure 5 Photographs of ITO electrodes after application of DC 5.0 V in the rubrene suspension containing SDS for various periods.

bottom edge when the application time was 5 minutes or less, suggesting that the flocculation as well as sedimentation of colloidal particles take place before the deposition.

The results of the voltage reduction are shown in Fig. 6(a). It is shown that there is no effective deposition on the ITO electrode by the application of a voltage up to 4.0 V. On the other hand, a noticeable bubble generation occurred at the application of 4.0 V but not 3.0 V or below. Figure 6(b) shows the change in log(1/T) spectra of the suspension after application of various voltages. It was observed that log(1/T) relevant to the rubrene colloidal particles in the suspension gradually decreased with increasing voltage above 3 V. These results indicate that the electric current provided by the application of 3.0 to 4.0 V induces flocculation as well as sedimentation of rubrene colloidal particles without deposition on the ITO electrode.

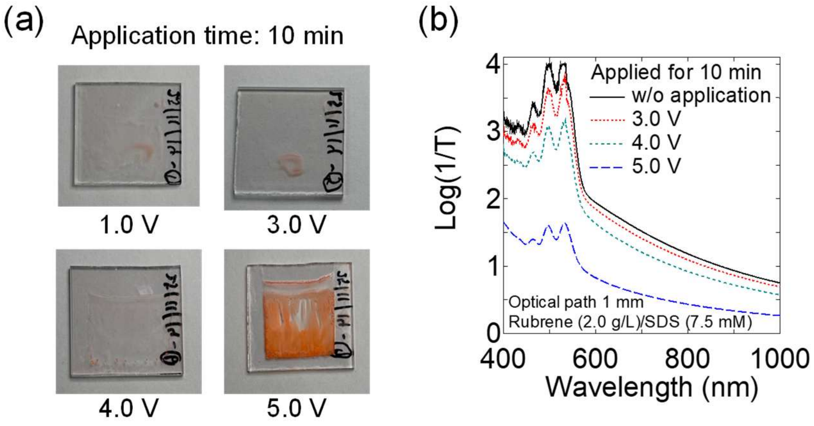

Figure 6 (a) Photographs of ITO electrodes after application of various voltages in the rubrene suspension containing SDS for 10 min. (b) Variation of log(1/T) spectrum of the suspension after DC voltage application for 10 min.

To summarize, with this aqueous colloidal suspension containing the surfactant SDS, application of insufficient voltage or application time results in partial coverage of the

ITO electrode, accompanied by flocculation and sedimentation of colloidal particles. From this observation, we can build a hypothesis that the application of voltage induces not only the deposition of colloidal particles on the oppositely charged electrode, but also their flocculation and sedimentation, and the latter is probably dominant at low voltage. If so, it seems natural to expect that the former becomes dominant as the voltage increases.

As shown in Fig. 7(a), the application of 15 V for several tens of seconds resulted in films that entirely cover the electrodes in contact with the suspension. The changes in the log(1/T) spectrum of the suspension due to the deposition at 15.0 V are shown in Fig. 7(b). The reduction of log (1/T) by application of 15.0 V for 10 s is very similar to that by 4.0 V for 10 minutes, shown in Fig. 6(b), while the former was accompanied by film deposition and the latter was not, confirming the high validity of the hypothesis and expectation mentioned above. The monotonic reduction in log(1/T) with the application time indicates that the thickness of the film is easy to control.

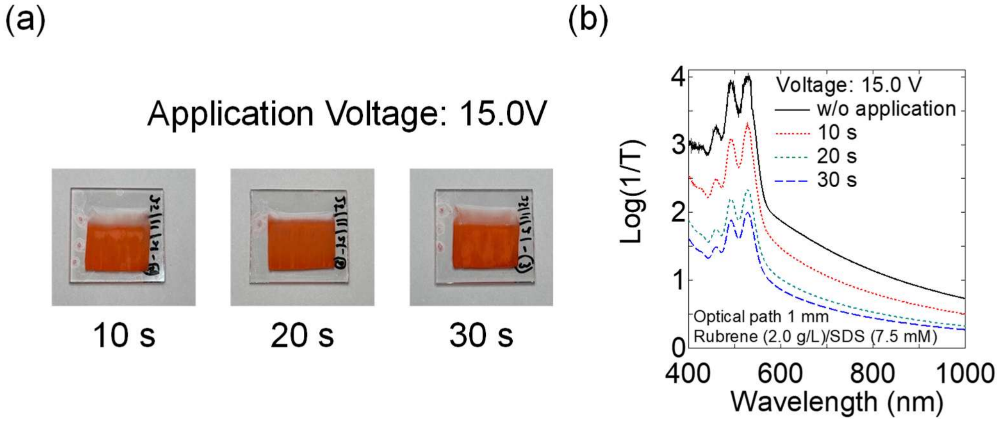

Figure 7 (a) Photographs of ITO electrodes after application of DC 15 V in the rubrene suspension containing SDS for various periods. (b) Variation of log(1/T) spectrum of the suspension after application of DC 15 V for various periods.

Figure 8 shows the optical absorption spectrum of the film deposited by application of 15 V for 10 s. Films prepared from colloidal suspensions by electric-field-induced deposition are essentially an accumulation of colloidal particles and are therefore inherently porous and exhibit light scattering. The spectrum has a relatively high baseline due to light scattering, but the optical absorption peaks characteristic of rubrene are clearly observed. Compared to the peaks in the optical absorption spectrum of a THF solution of rubrene, these peaks are red-shifted, a phenomenon commonly observed between solutions and solids of organic dyes.

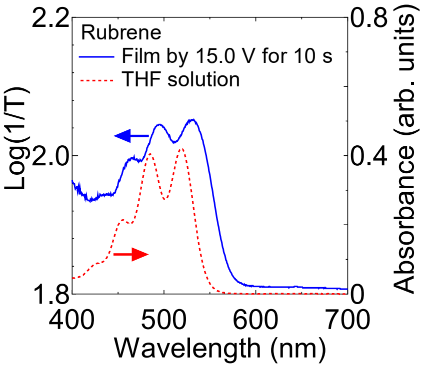

Figure 8 Log(1/T) spectrum of the rubrene film fabricated from an aqueous suspension containing SDS by application of DC 15.0 V for 10 s. Optical absorption spectrum of THF solution of rubrene (0.2 g/L) is also indicated for comparison.

Similar to films obtained by electrophoretic deposition, the rubrene films obtained by electric-field-assisted deposition in this study exhibit an inherently porous structure. A previous study showed that embedding in a porous polymer matrix is a promising way to enhance the photon upconversion characteristic of a rubrene-based molecular system. [27] The large surface area provided by the porosity is promising for electrochemical [28] and sensor [29] applications, other than optical applications such as photon upconversion films. On the other hand, if we target electronic devices such as organic light-emitting diodes and transistors, the porosity can deteriorate the carrier transport properties and should be removed. [30]

4. Conclusions

In this study, electric-field-induced deposition of rubrene from aqueous suspensions has been successfully demonstrated. Although the preparation of rubrene suspensions by reprecipitation using organic poor solvents was found to be difficult and not yet achieved, aqueous suspensions of rubrene suitable for electric-field-assisted deposition were obtained by injecting a small amount of THF solution of rubrene into aqueous solutions of SDS or DTAB. Aqueous solutions of SDS and DTAB, as poor solvents, result in negatively and positively charged rubrene colloidal particles, respectively. A systematic exploration of the deposition conditions revealed that the voltage required for uniform deposition of colloidal particles over the electrode is much higher than that required to initiate electrolysis of water, suggesting that the electric-field-assisted deposition achieved is not simple electrophoretic deposition but is strongly influenced by water electrolysis. These findings indicate that the deposition proceeds through a coupled electrophoretic–electrochemical process, potentially representing a new class of electric-field-assisted deposition process.

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All data generated or analyzed during this study are included in this published article

The authors report there are no competing interests to declare.

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