

## Research Article

# The Effects of Malonic Acid Derivatives and Acetic Acid Derivatives as Coadsorbents on the Photovoltaic Performance of Dye-Sensitized Solar Cells

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The effects of malonic acid derivatives and acetic acid derivatives as coadsorbents on the photovoltaic performance of D908 dye-sensitized nanocrystalline TiO<sub>2</sub> solar cells were investigated. Each of phenylmalonic acid (PMA) and cyclopentylacetic acid (CPEAA) coadsorptions was revealed to improve both the photocurrent and the photovoltage of the solar cells. The improved photocurrent was probably due to the suppression of self-quenching of the excited electrons in the dyes by coadsorption of PMA or CPEAA on the TiO<sub>2</sub> that increased in the electron-injection yields from the dye to the TiO<sub>2</sub>. The improved photovoltage was probably due to suppression of recombination between the injected electrons and I<sub>3</sub><sup>-</sup> ions on the TiO<sub>2</sub> surface. ATR-FTIR spectroscopy indicated that PMA or CPEAA coadsorption increased the content of bound dye on the TiO<sub>2</sub> surface. This result suggests that PMA or CPEAA coadsorption improved the photocurrent of the solar cells. Electrochemical impedance spectroscopy indicated that PMA or CPEAA coadsorption on the TiO<sub>2</sub> surface increased the charge recombination resistance (R<sub>2</sub>) and decreased the diffusion resistance in the electrolyte (R<sub>3</sub>). These results suggest that the coadsorption of PMA or CPEAA on the TiO<sub>2</sub> may improve its photovoltage and photocurrent.

## 1. Introduction

Dye-sensitized solar cells (DSCs) do not use high-purity silicon, and their manufacturing process is simple and of low cost. DSCs are expected to reduce the manufacturing cost significantly compared with conventional p-n junction solar cells.

A great deal of effort has been made to improve the photovoltaic performance of DSCs [1–4].

In our previous paper, we studied TiO<sub>2</sub> materials [5] and electrolytes [6].

The above study was found to be very important to control the interface between TiO<sub>2</sub> and dye electrolyte.

Dyes are required to be adsorbed on the TiO<sub>2</sub> in a monolayer without association or aggregation in order to suppress self-quenching of the excited electrons in the dyes.

To reduce association or aggregation of dyes, several carboxylic acid derivatives have been studied. For example, there are several reports using 3-phenylpropionic acid [7], deoxycholic acid [8], chenodeoxycholic acid [9], stearic acid [10], 4-guanidinobutyric acid [11], and hexadecylmalonic acid [12].

The coadsorption of dyes on the TiO<sub>2</sub> surface is expected to improve both the photocurrent and the photovoltage of DSC cells. The improved photocurrent can be attributed to a positive shift of the conduction band edge of TiO<sub>2</sub> [13] or to suppression of self-quenching of the excited electrons in the dyes [14]. The improved photovoltage can be caused by suppression of recombination between the injected electrons and I<sub>3</sub><sup>-</sup> ions [12, 13].

In this paper, we report the effects of malonic acid derivatives and acetic acid derivatives as coadsorbents on the

photovoltaic performance of DSC cells sensitized by D-908 dye (Figure 1).

Especially, dicarboxylic acids such as malonic acid derivatives could be anchored to the surface of  $\text{TiO}_2$  more effectively than monocarboxylic acids, and there are few previous papers about dicarboxylic acids as coadsorbents [12]. So, we choose malonic acids derivatives as coadsorbents.

## 2. Materials and Methods

**2.1. D908 Dye.** The molecular structure of D908 dye is shown in Figure 1.

D908 dye is one of the derivatives of Z907 dye. One of the protons bonded to the carboxylic groups is substituted by benzyltrimethylammonium cation.

D908 dye was purchased from Everlight Chemical Industrial Co., and used as supplied without further purification.

**2.2. Malonic Acid Derivatives and Acetic Acid Derivatives.** We chose six malonic acid derivatives including alkyl, cycloalkyl, and phenyl groups shown in Figure 2 and six acetic acid derivatives including alkyl, cycloalkyl, and naphthyl groups shown in Figure 3.

Isopropylmalonic acid (Tokyo Kasei Kogyo Co., Ltd.), n-butylmalonic acid (Wako Chemical Co., Ltd.), n-hexadecylmalonic acid (Oakwood Products, Inc.), cyclopentylmalonic acid (Tokyo Kasei Kogyo Co., Ltd.), phenylmalonic acid (Tokyo Kasei Kogyo Co., Ltd.), and benzylmalonic acid (Tokyo Kasei Kogyo Co., Ltd.) were used as supplied.

Cyclopropylacetic acid (Wako Chemical Co., Ltd.), cyclopentylacetic acid (Tokyo Kasei Kogyo Co., Ltd.), cyclohexylacetic acid (Tokyo Kasei Kogyo Co., Ltd.), cycloheptylacetic acid (Alfa Aesar), tert-butylacetic acid (Tokyo Kasei Kogyo Co., Ltd.), and 1-naphthylacetic acid (Tokyo Kasei Kogyo Co., Ltd.) were used as supplied.

**2.3. Preparation of  $\text{TiO}_2$  Electrodes Sensitized by D908 Dye.**  $\text{TiO}_2$  nanoparticle (ca. 20 nm) pastes were prepared by the method of the previous paper [15].  $\text{TiO}_2$  electrodes (apparent area  $0.25 \text{ cm}^2$ ; thickness ca  $14 \mu\text{m}$ ) were prepared on a glass substrate coated with F-doped  $\text{SnO}_2$  by screen printing. The electrodes were heated at  $550^\circ\text{C}$  for 1 hour. The D908 dye (0.5 mM) and the coadsorbent (0.5 mM) were dissolved in a 50 : 50 (vol.%) solution of acetonitrile and tert-butyl alcohol. The solvents were purchased from Kanto Chemical Co., Inc. and used as supplied without further purification. The  $\text{TiO}_2$  films were immersed in the dye solutions and kept at  $25^\circ\text{C}$  for 20 h.

**2.4. Preparation of Electrolyte.** The electrolyte consisted of 0.06 M iodine, 0.1 M LiI, 0.6 M 1-ethyl-3-methylimidazolium iodide, and 0.5 M 4-tert-butylpyridine in acetonitrile iodine (Wako Pure Chemical Industries, Ltd.), LiI (Kojundo Chemical Laboratory Co., Ltd.), 1-ethyl-3-methylimidazolium iodide (Kanto Chemical Co., Inc.), 4-tert-butylpyridine (Sigma-Aldrich), and acetonitrile (Kanto Chemical Co., Inc.) were used as supplied.

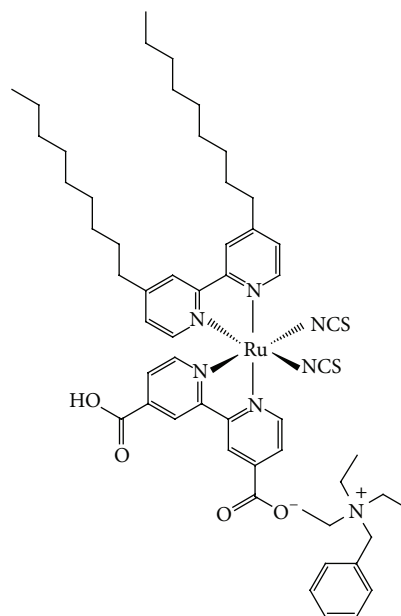


FIGURE 1: Structure of D908 dye.

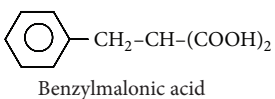
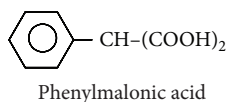
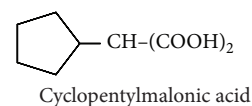
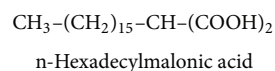
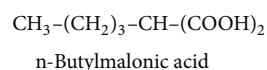
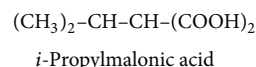


FIGURE 2: Structures of malonic acid derivatives.

**2.5. Fabrication of DSC Cells and Photovoltaic Measurement.** The DSC cell consisted of the  $\text{TiO}_2$  electrode, a counter electrode, a polyimide film spacer ( $35 \mu\text{m}$  thick), and the electrolyte. The counter electrode was a Pt film sputtered on a TCO-coated glass plate.

Photovoltaic measurement employed an AM1.5 solar simulator equipped with a xenon lamp (San-ei Electric Co., Ltd.).

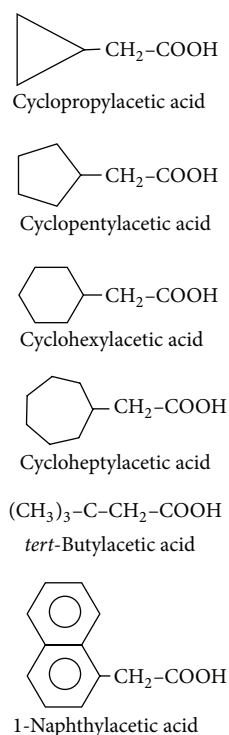


FIGURE 3: Structures of acetic acid derivatives.

**2.6. Measurement of the Amount of Dye Adsorbed on  $\text{TiO}_2$ .** The areas around the  $\text{TiO}_2$  films sensitized by D908 dye were wiped with ethanol. Then, the  $\text{TiO}_2$  films were immersed in a 1.5 : 1.0 (vol.%) solution of ethanol and 0.1 M KOH for 10 minutes. After recognizing the dye desorption, 1.0 mL of the solution was used to measure its specific adsorption (maximum adsorption wavelength: 510 nm) by the spectrophotometer (Shimadzu UV-2550). The amounts of adsorbed dyes were calculated from the absorbance of 510 nm light for 1.0 mL desorbed sample solutions.

**2.7. Measurement of Attenuated Total Reflection Fourier-Transformed IR (ATR-FTIR) Spectroscopy.** The surfaces of  $\text{TiO}_2$  films sensitized by D908 dye, D908 dye with phenylmalonic acid, and D908 dye with cyclopentylacetic acid were analyzed by ATR-FTIR (Nicolet, iN10MX) with 128 scans at a resolution of  $8 \text{ cm}^{-1}$ .

**2.8. Measurement by Raman Spectroscopy.** The surfaces of  $\text{TiO}_2$  films sensitized by D908 dye, D908 dye with phenylmalonic acid, and D908 dye with cyclopentylacetic acid were analyzed with a Raman microscope (Renishaw, inVia) with an exciting light of 532 nm wavelength, an exciting light power of 0.5% (0.35 mW), an objective mirror of 50x magnification, an irradiation time of 3 seconds, 180-degree back scatter, and 5 times of scanning. The wave number was calibrated by silicone.

**2.9. Measurement by Electrochemical Impedance Spectroscopy (EIS).** The large DSC cells using the  $\text{TiO}_2$  electrodes (apparent area  $5.1 \text{ cm}^2$ , thickness ca  $14 \mu\text{m}$ ) sensitized by D908 dye, D908 dye with phenylmalonic acid, and D908 dye with cyclopentylacetic acid were fabricated and measured with an electrochemical impedance spectroscope (EIS) (Biologic, SP-300) under irradiation of  $100 \text{ mW/cm}^2$  at  $25^\circ\text{C}$ .

### 3. Results and Discussion

**3.1. Photovoltaic Performance.** Table 1 shows the amount of D908 dyes adsorption and the photovoltaic parameters of DSC devices without coadsorbent (control) and with isopropylmalonic acid (IPMA), n-butylmalonic acid (BMA), n-hexadecylmalonic acid (HDMA), cyclopentylmalonic acid (CPMA), phenylmalonic acid (PMA), and benzylmalonic acid (BZMA).

As shown in Table 1, all the adsorbed amounts of the D908 dyes decreased when the six types of malonic acid derivatives were used as coadsorbents. These results indicate that malonic acid derivatives were adsorbed on the  $\text{TiO}_2$  and suppressed D908 dye adsorption.

For the DSC device with HDMA, the amounts of the adsorbed D908 dyes dramatically decreased to  $11.9 \times 10^{-8} \text{ mol/cm}^2$  relative to the control device ( $19.4 \times 10^{-8} \text{ mol/cm}^2$ ). This result suggests that HDMA significantly suppressed D908 dye adsorption by its bulky steric effects. As a result of decreased dye adsorption, the value of  $J_{sc}$  decreased from 16.4 to 14.8  $\text{mA/cm}^2$ , and the value of conversion efficiency also decreased from 6.9% to 6.6%.

The other five DSC devices improved their conversion efficiencies. In particular, the DSC device with PMA showed the highest value of conversion efficiency (7.8%) among all the seven DSC devices. The amount of the adsorbed D908 dye decreased to  $18.1 \times 10^{-8} \text{ mol/cm}^2$  relative to the control device ( $19.4 \times 10^{-8} \text{ mol/cm}^2$ ), but the device showed a 6% improvement in the value of  $J_{sc}$  (17.4  $\text{mA/cm}^2$ ) relative to that of the control device (16.4  $\text{mA/cm}^2$ ). This result suggests that the coadsorption of PMA on the  $\text{TiO}_2$  suppressed the aggregation of the dyes and the self-quenching of the excited electrons in the dyes by its steric effect. The effect is described in Section 3.2 ATR-FTIR spectroscopy and Section 3.4 electrochemical impedance spectroscopy. Therefore, the coadsorption of PMA increased in electron-injection yields from the dye to the  $\text{TiO}_2$ .

The device also showed a 3% improvement in  $V_{oc}$  (0.66 V) relative to the control device (0.64 V). This result suggests that the coadsorption of PMA on the  $\text{TiO}_2$  suppressed the recombination between the injected electrons and  $\text{I}_3^-$  ions. The effect is also described in Section 3.4 electrochemical impedance spectroscopy.

Table 2 shows the amounts of the adsorbed D908 dyes and the photovoltaic parameters of DSC devices with cyclopropylacetic acid (CPRAA), cyclopentylacetic acid (CPEAA), cyclohexylacetic acid (CHEAA), cycloheptylacetic acid (CHPAA), tert-butylacetic acid (TBAA) and naphthylacetic acid (NPAA). As shown in Table 2, all the amounts of the adsorbed D908 dyes decreased when the six types of

TABLE 1: Photovoltaic parameters of DSC devices with malonic acid derivatives.

Coadsorbent	The amount of dye adsorption $10^{-8}$ mol/cm <sup>2</sup>	$J_{sc}$ (mA/cm <sup>2</sup> )	$V_{oc}$ (V)	FF	$\eta$ (%)
Control	19.4	16.4	0.64	0.66	6.9
Isopropylmalonic acid	13.9	16.3	0.64	0.69	7.2
n-Butylmalonic acid	14.1	15.8	0.64	0.69	7.0
n-Hexadecylmalonic acid	11.9	14.8	0.64	0.70	6.6
Cyclopentylmalonic acid	14.8	16.7	0.64	0.68	7.3
Phenylmalonic acid	18.1	17.4	0.66	0.68	7.8
Benzylmalonic acid	12.6	16.4	0.63	0.68	7.0

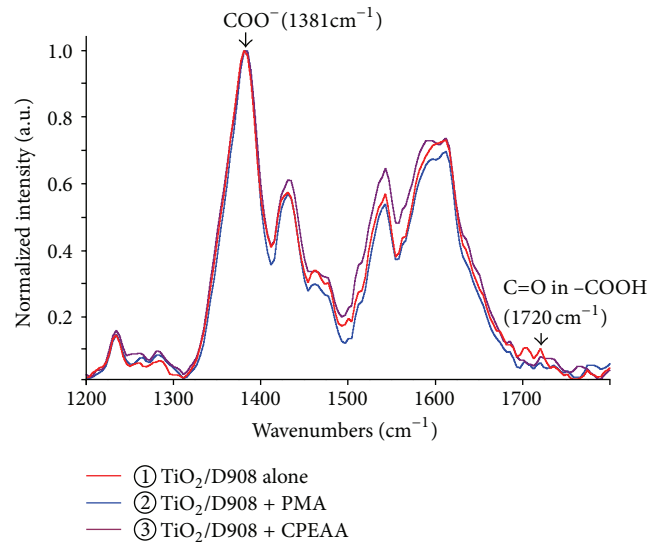
TABLE 2: Photovoltaic parameters of DSC devices with acetic acid derivatives.

Coadsorbent	The amount of dye adsorption $10^{-8}$ mol/cm <sup>2</sup>	$J_{sc}$ (mA/cm <sup>2</sup> )	$V_{oc}$ (V)	FF	$\eta$ (%)
Control	19.4	16.4	0.64	0.66	6.9
Cyclopropylacetic acid	17.1	16.6	0.62	0.67	6.9
Cyclopentylacetic acid	17.2	17.0	0.66	0.68	7.6
Cyclohexylacetic acid	18.2	17.0	0.64	0.67	7.3
Cycloheptylacetic acid	17.3	16.5	0.62	0.67	6.9
tert-Butylacetic acid	18.7	16.6	0.63	0.68	7.1
1-Naphthylacetic acid	18.0	16.3	0.61	0.68	6.8

acetic acid derivatives were used as coadsorbents. These results suggest that acetic acid derivatives were adsorbed on the TiO<sub>2</sub> and suppressed D908 dye adsorption. Three DSC devices with CPEAA, CHEAA, or TBAA improved their conversion efficiencies. In particular, the device with CPEAA showed the highest value of conversion efficiency (7.6%) among seven devices. The amount of adsorbed D908 dye decreased to  $17.2 \times 10^{-8}$  mol/cm<sup>2</sup> relative to the control device ( $19.4 \times 10^{-8}$  mol/cm<sup>2</sup>), but the device showed a 4% improvement in  $J_{sc}$  (17.0 mA/cm<sup>2</sup>) relative to the control device (16.4 mA/cm<sup>2</sup>). The device also showed a 3% improvement in  $V_{oc}$  (0.66 V) relative to the control device (0.64 V). These results suggest that the coadsorption of CPEAA on the TiO<sub>2</sub> has an effect similar to that of PMA. The effect is also described in Section 3.2 ATR-FTIR spectroscopy and Section 3.4 Electrochemical impedance spectroscopy.

**3.2. ATR-FTIR Spectroscopy.** Figure 4 shows the attenuated total reflection Fourier-transformed IR (ATR-FTIR) spectra of TiO<sub>2</sub> films sensitized by D908 alone, D908 with PMA, and D908 with CPEAA in the range of 1200–1800 cm<sup>-1</sup>. The assignments of the peaks were prepared by the previous papers [10, 16].

The peak at 1381 cm<sup>-1</sup> was assigned to the carboxylate groups (-COO<sup>-</sup>) anchored to the surface of the TiO<sub>2</sub>, and the peak at 1720 cm<sup>-1</sup> was assigned to the free carboxylic acid (-COOH) of the D908 dye. The intensity at 1381 cm<sup>-1</sup> relative to that at 1720 cm<sup>-1</sup> ( $I_{1381}/I_{1720}$ ) was calculated, and the values were 11.2 (D908 alone, control), 21.3 (D908 with PMA), and 17.2 (D908 with CPEAA), respectively.

FIGURE 4: ATR-FTIR spectra of TiO<sub>2</sub> films sensitized with D908 alone, D908 with PMA, and D908 with CPEAA.

Therefore, the amounts of D908 dyes anchored to the surface of TiO<sub>2</sub> were in the following order: D908 with PMA > D908 with CPEAA > control. This result provides an explanation for improved  $J_{sc}$  value for the DSC device with PMA or CPEAA, even though the amounts of dye were less.

**3.3. Raman Spectroscopy.** Figure 5 shows the Raman spectra of TiO<sub>2</sub> films sensitized by D908 alone, D908 with PMA, and D908 with CPEAA in the range of 400–1800 cm<sup>-1</sup>. The

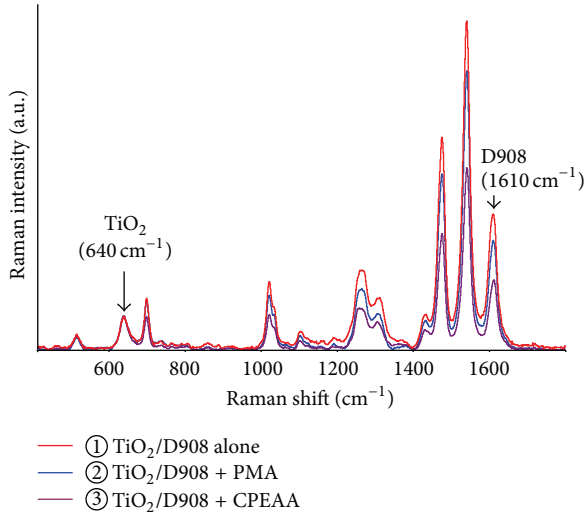


FIGURE 5: Raman spectra of the  $\text{TiO}_2$  films sensitized with D908 alone, D908 with PMA, and D908 with CPEAA.

assignments of the peaks were prepared by the previous paper [17].

The peak at  $640\text{ cm}^{-1}$  was assigned to the  $\text{TiO}_2$ , and the peak at  $1610\text{ cm}^{-1}$  was assigned to the D908 dye. The intensity at  $1610\text{ cm}^{-1}$  relative to that at  $640\text{ cm}^{-1}$  ( $I_{1610}/I_{640}$ ) was calculated, and the values were 4.26 (D908 alone, control), 3.26 (D908 with PMA), and 2.03 (D908 with CPEAA), respectively.

Therefore, the amounts of D908 dye adsorbed on the surface of  $\text{TiO}_2$  were in the following order: control > D908 with PMA > D908 with CPEAA. This result coincides with the order of the amounts of D908 dyes measured by the spectrophotometer as referred to in Section 2.7, and suggests that Raman spectra can be used for the evaluation of the amount of dye more conveniently.

**3.4. Electrochemical Impedance Spectroscopy (EIS).** Electrochemical impedance spectroscopy (EIS) was performed to investigate the interface in the DSC devices by the method of the previous papers [18, 19].

Figure 6 shows the Nyquist plots of DSC devices using the  $\text{TiO}_2$  films sensitized by D908 alone, D908 with PMA, and D908 with CPEAA. The plots were fitted by the equivalent circuit of DSC shown in Figure 7, and  $R_s$ ,  $R_1$ ,  $R_2$ , and  $R_3$  values were calculated.

$R_s$  means the series resistance such as that of F-doped  $\text{SnO}_2$  and interconnection.

$R_1$  (Pt) means the resistance of charge transfer between Pt and electrolyte.  $R_2$  ( $\text{TiO}_2$ ) means the charge recombination resistance from  $\text{TiO}_2$  to  $\text{I}_3^-$  ions.  $R_3$  (I) means the diffusion resistance in electrolyte. These parameters are shown in Table 3.

The values of  $R_2$  in the DSC devices with PMA or CPEAA were larger than that of the control device. This result suggests that the coadsorption of PMA or CPEAA on the  $\text{TiO}_2$  suppressed the recombination between the

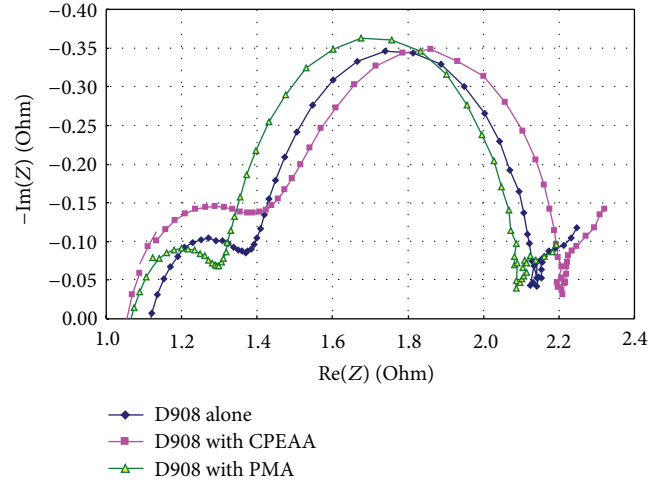


FIGURE 6: Nyquist plots for the DSC devices using  $\text{TiO}_2$  films sensitized with D908 alone, D908 with PMA, and D908 with CPEAA.

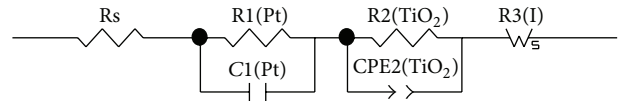


FIGURE 7: Equivalent circuit of DSC.

TABLE 3: Impedance parameters of DSC devices with PMA or CPEAA.

Coadsorbent	$R_s$	$R_1$ (Pt)	$R_2$ ( $\text{TiO}_2$ )	$R_3$ (I)
Control	5.5	1.2	3.8	3.6
Phenylmalonic acid	5.5	1.2	4.0	3.4
Cyclopentylacetic acid	5.5	1.2	4.0	3.4

injected electrons and  $\text{I}_3^-$  ions; therefore, PMA or CPEAA coadsorption improved the  $V_{oc}$  of the DSC devices.

The values of  $R_3$  in the DSC devices with PMA or CPEAA were smaller than that of the control device. This result suggests that the coadsorption of PMA or CPEAA on the  $\text{TiO}_2$  suppressed the aggregation of the dyes and  $\text{I}^-$  ions were able to access the dyes more smoothly; thus PMA or CPEAA coadsorption improved the  $J_{sc}$  of the DSC devices.

## 4. Conclusions

Coadsorption of PMA or CPEAA improved both the  $J_{sc}$  and the  $V_{oc}$  values for DSC devices sensitized by D908 dye even though coadsorption decreased the amount of dye adsorbed on the  $\text{TiO}_2$  electrode. The conversion efficiency increased from 6.9% to 7.8% for PMA and from 6.9% to 7.6% for CPEAA.

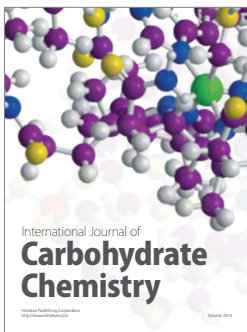
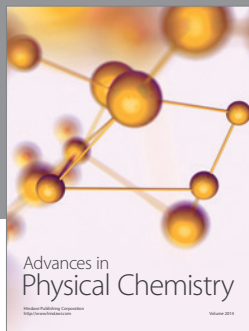
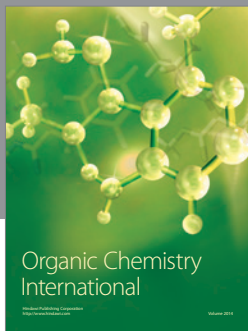
ATR-FTIR spectra suggested that coadsorption of PMA or CPEAA increased the amount of D908 dye anchored to the surface of  $\text{TiO}_2$ . This result provides an explanation for the improved  $J_{sc}$  values.

Raman spectra suggested that coadsorption of PMA or CPEAA decreased the amount of D908 dye adsorbed on the surface of TiO<sub>2</sub>. This result coincides with the amount of D908 dye measured by the spectrophotometer and suggests that Raman spectra can be used for the evaluation of the amount of dye more conveniently.

Electrochemical impedance spectroscopy suggested that the values of R<sub>2</sub> in the DSC devices with PMA or CPEAA were larger than that of the control device. This result suggests that the coadsorption of PMA or CPEAA on the TiO<sub>2</sub> suppressed the recombination between the injected electrons and I<sub>3</sub><sup>-</sup> ions, PMA or CPEAA coadsorption improving the V<sub>oc</sub> of the DSC devices. The values of R<sub>3</sub> in the DSC devices with PMA or CPEAA were smaller than that of the control device. This result suggests that the coadsorption of PMA or CPEAA on the TiO<sub>2</sub> suppressed the aggregation of the dyes and I<sup>-</sup> ions were able to access the dyes more smoothly. Consequently, PMA or CPEAA coadsorption improved the J<sub>sc</sub> of the DSC devices.

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