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Formation of MUA (mercaptoundeconic acid)-capped CDSe nanoparticle films by electrophoretic deposition

Khatijah A. Yaacob*,[1], D. Jason Riley

Imperial College London, Department of Materials, Exhibition Road, SW7 2AZ London, UK

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Abstract

Electrophoretic deposition (EPD) has gained increasing interest for the deposition of materials such as TiO_2, carbon nanotubes and trioctylphosphine oxide (TOPO)-capped CdSe nanoparticles. In this study, a mercaptoundecanoic acid (MUA) CdSe nanoparticle film was formed by electrophoretic deposition. A colloidal suspension of TOPO-capped CdSe nanoparticles was prepared by the hot injection method, followed by ligand exchange to produce MUA-capped CdSe nanoparticles. As-prepared MUA-capped CdSe nanoparticles were washed using ethyl acetate and ethyl ether. Then, the washed nanoparticles were resuspended in ethanol and immediately used for EPD. A CdSe nanoparticle film measuring 2.75 μm in thickness was deposited at an applied voltage of 5 V and deposition time of 5 min.

Keywords: Mercaptoundeconic acid (MUA)-capped CdSe; Trioctylphosphine oxide (TOPO)-capped CdSe; Ligand exchange; Electrophoretic deposition

1. Introduction

Electrophoretic deposition has recently been used to assemble nanoparticles on electrodes to create useful nanostructures. Electrophoretically deposited CdSe powders on SnO_2-coated glass substrates were studied by Ueno and co-workers [1] in 1983. The researchers were able to produce smooth films with well-adhered deposits on the glass substrates.

The electrophoretic deposition of CdSe nanoparticles was first described by Islam and co-worker [2] in 2002. Their paper describes the electrodeposition of patterned CdSe nanoparticle films on Si wafers coated with a 10 nm layer of Ti followed by a ~150 nm layer of Au. The work concludes that electrophoretic deposition can be used to deposit nanoparticles and that it is feasible to deposit nanoparticles in patterns. To form high-quality electrophoretic CdSe films, there are two critical procedures that must be followed. The first procedure is the repeated washing of CdSe nanoparticles from a fresh solution to remove the ligands, which makes the nanoparticles highly charged and hence more strongly adherent to substrates [3–5]. The second procedure is the treatment of the films after electrodeposition to make the films chemically stable [3,4].

Kamat and co-worker [6] also used electrophoretic deposition to deposit CdSe–nC_{60} nanoparticles on thin films of SnO_2 cast on optically transparent electrodes (OTEs). The CdSe and nC_{60} particles were mixed in specified ratios and suspended in a mixed toluene/acetonitrile solvent; under an applied field of 50 V cm^{-1}, CdSe–nC_{60} clusters were deposited on a positively charged electrode. The IPCE obtained from the CdSe–nC_{60} electrodes was ~4%.

Rosenthal and co-workers [7] fabricated photovoltaic cells by the electrophoretic deposition of CdSe nanoparticles suspended in hexanes on TiO_2/indium tin oxide (ITO) substrates. The TiO_2 layer was formed by spin coating onto an ITO/ glass substrate, followed by the electrophoretic deposition of TOPO-capped CdSe suspend in hexanes at an applied voltage of 500 V. Very low conversion efficiency was observed from a solar cell fabricated by the electrophoretic deposition of CdSe nanocrystals because a very thin CdSe film was deposited on the TiO_2/ITO substrate.

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*Corresponding author. Tel.: +60 4599 5267; fax: +60 4594 1011.
E-mail address: mraisha@eng.usm.my (K.A. Yaacob).

[1] Present address: School of Materials and Mineral Resources Engineering, Universiti Sains Malaysia, Engineering Campus, 14300 Nibong Tebal, Pulau Pinang, MALAYSIA

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Zaban and co-workers [8] reported the fabrication of quantum dot-sensitised solar cells by the electrophoretic deposition of CdSe quantum dots onto conducting electrodes coated with mesoporous TiO_2. Mesoporous TiO_2 was also prepared by the electrophoretic deposition of Degussa P25 particles onto fluorine-doped tin oxide (FTO), followed by hydraulic pressing and sintering at 550 °C for 1 h. The mesoporous TiO_2 electrodes were then immersed in a suspension of CdSe nanoparticles in toluene, and electrophoretic deposition was performed at an applied voltage of 200 V for 2 h. The incident photon-to-electron conversion efficiencies show an incident photon-to-current efficiency (IPCE) in the range of 13–20% for all particle sizes, with larger particles exhibiting a longer onset wavelength corresponding to their smaller band gap energies.

Recently, Sakthi and his co-workers [9] have combined molecular linker approaches and electrophoretic deposition in order to deposit CdSe nanoparticles suspended in chloroform onto TiO_2 electrode. From their results, a layer of CdSe thin film was successfully deposited at 30 V within 15 min deposition time. Zillner et al. [10] used electrophoretic deposition to form a heterojunction of CdTe/CdSe nanoparticles to be applied in fabrication of nanodevices. Their results shows that the deposited layer with the thickness of 100–300 nm were deposited at applied voltage of 500 V for 8 min deposition time. The thickness layers can be controlled by changing the concentration of nanoparticles in the suspension. Besides using electrophoretic deposition of CdSe nanoparticles in fabricating quantum dots sensitised photoanode, this technique was also used in fabricating light emitting diode [11] and biosensor [12].

In all of the electrophoretic studies discussed above, CdSe nanoparticles were suspended in non-polar solvents, e.g., hexane and toluene. Electrophoretic deposition from a non-polar solvent normally requires higher voltages (>30 V) or longer deposition times (>15 min). In this study, CdSe nanoparticles were suspended in a polar solvent, e.g., ethanol, and were electrophoretically deposited on a fluorine-doped indium tin oxide (FTO) substrate. Our paper is the first to report the electrophoretic deposition of CdSe nanoparticles suspended in a polar solvent. The results show that this process is feasible for large-scale processing in the future due to the short fabrication time.

2. Experimental

2.1. Synthesis of MUA capped CdSe

Eight grams of trioctylphosphine oxide, 5.0 g of hexadecylamine, and 0.15 g of tetradecylphosphine acid were mixed in a three-neck flask. The mixture was dried and degassed under vacuum at 110 °C for 1 hr 30 min with the periodic flushing of argon. After 90 min, the flask was placed under an Ar atmosphere, and at 110 °C a TOPSe (trioctylphosphine Se) stock solution, prepared in a dry box by dissolving 0.158 g of Se powder in 2.0 ml TOP, was injected into the flask. The temperature was then raised to 300 °C. A TOPCd stock solution prepared in a dry box by adding 0.120 g of cadmium acetate to 3 ml of TOP was rapidly injected into the mixture under vigorous stirring, resulting in a yellow-orange solution. When the required particle size was achieved, the flask was removed from the heater, and the reaction was quenched in toluene. The excess capping agents were removed by precipitating the CdSe particles in methanol, centrifuging the suspension at 5500 rpm for 10 min and resuspending the particles in toluene. In this study, the method of Peng et al. [13] for ligand exchange from trioctylphosphine oxide (TOPO)-capped CdSe particles to mercaptoundecanoic acid (MUA)-capped CdSe particles was employed. All chemicals were purchased from Sigma-Aldrich and were used as received.

2.2. Electrophoretic deposition

The set-up used for the electrophoretic deposition of MUA-capped CdSe nanoparticles consisted of a power supply and digital voltmeter. The digital voltmeter was connected in parallel to the electrodes and the power supply. Because ethanol is a polar solvent, only a DC voltage in the range 5–20 V was applied across the electrode. The DC current was monitored using the voltmeter.

2.3. Characterisations

Absorption spectra were obtained using a Perkin Elmer model Lambda Bio 10 UV visible spectrophotometer with a spectral resolution of 2 nm. Photoluminescence (PL) spectra were recorded over the range of 350–650 nm using an excitation wavelength of 340 nm and integration time of 0.1 s on a Photon Technology International (PTI) fluorescence instrument. Inductively couple plasma optical emission spectroscopy (ICP-OES) was used to quantitatively analyse the elements in solution after washing. Zeta potential measurements were carried out using a ZetaPALS (phase analysis light scattering) zeta potential analyser from the Brookhaven Instrument Corporation, USA. A Zygo® New View white-light microscope-based interferometer equipped with the MetroPro software programme was employed to measure the thickness of the MUA-capped CdSe layers on FTO.

The photocurrents were recorded at room temperature using a custom-made three-electrode cell consisting of Ag/AgCl/KCl as the reference electrode, a curled platinum mesh as the counter electrode and a layer of semiconductor nanoparticles deposited on an FTO substrate as the working electrode. The cell voltage was controlled and the current measured by a home-built potentiostat. A 75 W Xenon short arc lamp housed in a LPS-220B PTi lamp power supply in conjunction with a PTi SID-101 monochromator controller was used as a light source to illuminate the samples. The photocurrent was recorded as a function of incident wavelength using customised software. The recorded photocurrent on the working electrode can be converted into incident photon-to-current efficiency (IPCE) by calculating the ratio of the number of electrons successfully converted into photocurrent to the number of incident photons radiated from the lamp.

3. Results & discussion

3.1. Optical properties of MUA capped CdSe

The absorption spectra of TOPO-capped CdSe nanoparticles before and after ligand exchange are shown in Fig. 1a. The figure shows a broad absorption peak due to the wide size distribution of CdSe nanoparticles in the solution. In this experiment, all solutions were diluted to exhibit the same absorbance peak. The MUA-treated CdSe nanoparticles maintained their original spectrum, with the position and the width of the absorption peak remaining unchanged even after one purification cycle (Fig. 1a). This result suggests that no oxidation, growth, etching or aggregation of the nanoparticles occurred during the exchange process [14]. The diameter of these CdSe nanoparticles was calculated to be 3.26 nm from the absorption peak of 560 nm using the following equation provided by Peng et al. [13]:

$$D=(1.6122\times10^{-19})\lambda^4-(2.6575\times10^{-6})\lambda^3+(1.6242\times10^{-3})\lambda^2-(0.4277)\lambda+41.57 \qquad (1)$$

where D is the average diameter (nm) of the CdSe nano particles and λ is the wavelength (nm) of the absorption peak.

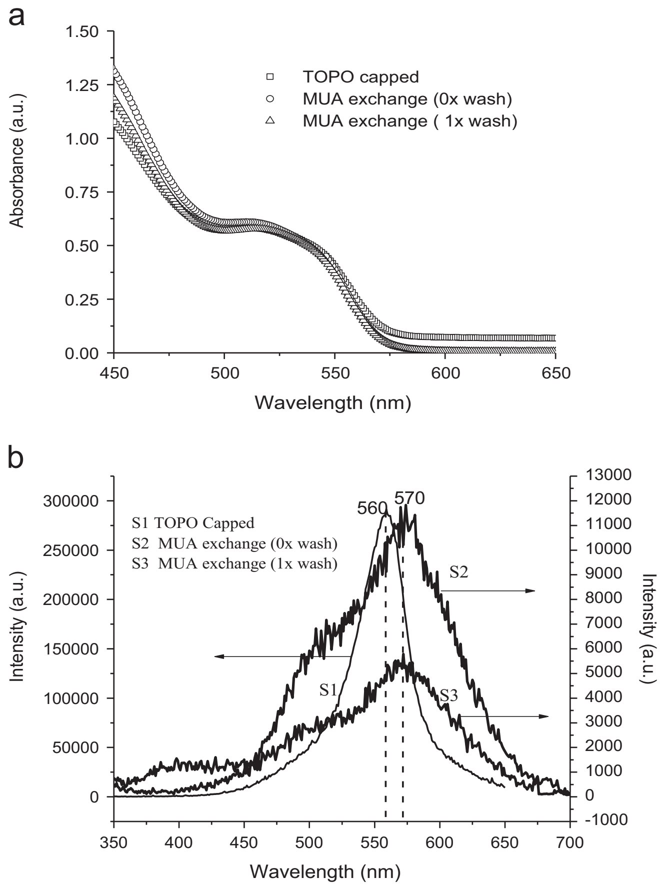

Fig. 1. (a) Absorption spectra and (b) PL spectra of original TOPO-capped CdSe nanoparticles, fresh MUA-capped CdSe nanoparticles and MUA-capped CdSe nanoparticles submitted to a single wash cycle.

As shown in Fig. 1b, the maximum PL peak intensity of the unpurified MUA-capped CdSe nanoparticles was reduced by approximately one order of magnitude relative to that of the TOPO-capped CdSe nanoparticles, and a red shift of approximately 10 nm was observed. The reduction in intensity was probably caused by the poor surface termination of the MUA-capped CdSe nanoparticles: when ligands are exchanged for more-polar ligands, PL intensity is typically reduced. This red shift may occur for two reasons. The first is the Ostwald ripening of the CdSe nanoparticles that occurs during the ligand exchange process. Small particles dissolve, and the monomers released are consumed by large particles. The second is that the dielectric of MUA differs from that of TOPO. It is difficult to determine which effect is more responsible for the red shift in the PL spectra of the MUA-capped CdSe.

Fig. 1b also shows the PL peak intensity of the MUA-capped CdSe nanoparticles after one washing cycle. Following the MUA treatment, TOPO-capped CdSe nanoparticles, were precipitated from the methanol suspension by the addition of the same amount of ethyl acetate and ethyl ether. The supernatant was removed, and the nanoparticles were resuspended in ethanol for electrophoretic deposition. After the washing procedure, the intensity of the PL spectra was reduced. The PL spectra after washing reflect the removal of some surface-bound MUA ligands. The normalised PL spectra recorded before and after the washing procedure show good overlap and a shift in the maximum peaks. The binding of MUA to CdSe was found to be quite strong, as the bound MUA molecules were retained even after one washing cycle. Further washing resulted in agglomeration.

3.2. EPD of MUA-capped CdSe

3.2.1. Applied voltage during EPD of MUA-capped CdSe suspension

In this research, we used three different suspension media for electrophoretic deposition: water, methanol and ethanol. Water was the first suspension medium used for the EPD process at applied voltages of 1–5 V. The results show that deposition of CdSe occurred at an applied voltage of 5 V; at lower potentials, no deposition was observed. From Gibbs free energy, the hydrolysis of pure water occurs at 1.23 V. However, in this research the hydrolysis of water begins to occur at 5 V, as reported by Van der Biest & Vandeperre [15]. This is due to addition of tetramethylammonium hydroxide (TMAH) to the suspension in order to deprotonate the COOH groups of MUA.

For ethanol and methanol, the voltages applied were between 2.5 V and 20 V. Good deposition layers were obtained at an applied voltage of 5 V for nanoparticles dispersed in ethanol. At 10 V, the deposits obtained from each alcohol became thicker and peeled off due to poor adhesion to the substrate. At a higher voltage of 20 V, the initial current was also high and tended to burn the cathode. Moreover, due to the high voltage applied, the suspensions in methanol and ethanol were susceptible to Joule heating, which sometimes also led to the loss of stability of the

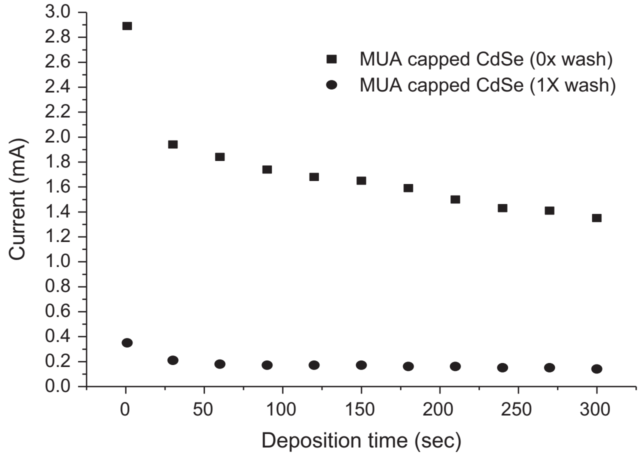

Fig. 2. EPD current intensity vs. deposition time for unpurified and washed (single cycle) MUA-capped CdSe nanoparticles in methanol.

Table 1
ICP-OES results for MUA-capped CdSe nanoparticles: purified and after one wash cycle.

No. of wash	Cd (ppm)	Se (ppm)	S (ppm)	P (ppm)	Cd/Se
0 ×	8121	4718	6.638	35.36	1.72
1 ×	8880	12140	3.845	30.28	0.73

suspensions. In such cases, the MUA-capped CdSe nanoparticles sedimented to the bottom of the EDP cell during EPD. In future research, ethanol will be used as the suspension medium for MUA-capped CdSe nanoparticles and an applied voltage of 5 V will be used.

3.2.2. Apparent pH of MUA-capped CdSe suspension

The apparent pH of MUA-capped CdSe sols is one of the important parameters that must be considered in electrophoretic deposition. The apparent pH of MUA-capped CdSe sols is required to be greater than 10 to fully deprotonate the COOH groups of MUA and yield negatively charged COO^- surface groups. The negative charge distributed on the surface will stabilise the nanoparticles. The zeta potential of the nano particle suspension at apparent pH 11 was −23.66 ± ± 2.1 mV. This value indicates that the nanoparticles were negatively charged. Under an applied electric field, the nanoparticles drifted toward the anode.

3.2.3. Purification of MUA-capped CdSe suspension

In this experiment, washing the as-prepared MUA-capped CdSe nanoparticles is important in removing any unincorporated MUA and excess TMAH from the ligand exchange process. Impurities enhance the conductivity of the solution and affect the current intensity during electrophoretic deposition. Fig. 2 shows a plot of current intensity vs. deposition time during the EPD of 1.45×10^{15} dots cm^{-3} at apparent pH 11 for both unpurified and washed (single cycle) MUA-capped CdSe nanoparticles. A voltage of 5 V was applied for 5 minutes. The current intensity used to deposit the unpurified MUA-capped CdSe nanoparticles was higher than that used to deposit the washed MUA-capped CdSe nanoparticles. This demonstrates that the unpurified particles had more charge carriers due to excess deprotonated MUA. During ligand exchange, the MUA powder tends to deprotonate in the presence of TMAH. Under an applied voltage, the deprotonated MUA will deposit on the anode but not strongly adhere to it. After drying, the deposited film tends to peel off. The ICP results for sulphur shown in Table 1 confirm that more MUA occurred in the unpurified sols than in the washed sols, where sulphur was derived from the thiol group at the head of MUA.

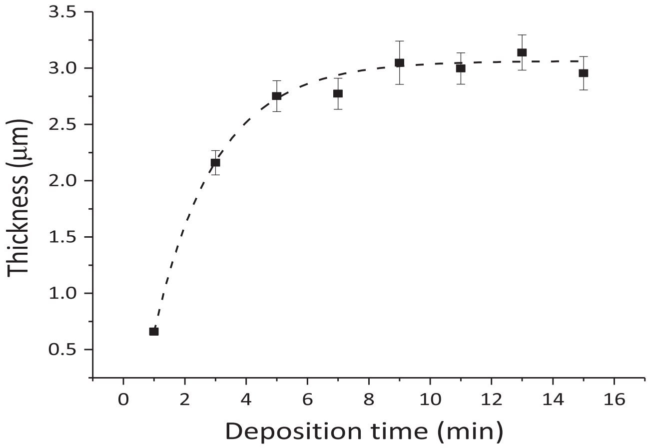

Fig. 3. Thickness of MUA-capped CdSe nanoparticles in a concentration of 3.67×10^{15} dots cm^{-3} at different deposition time under an applied voltage of 5 V.

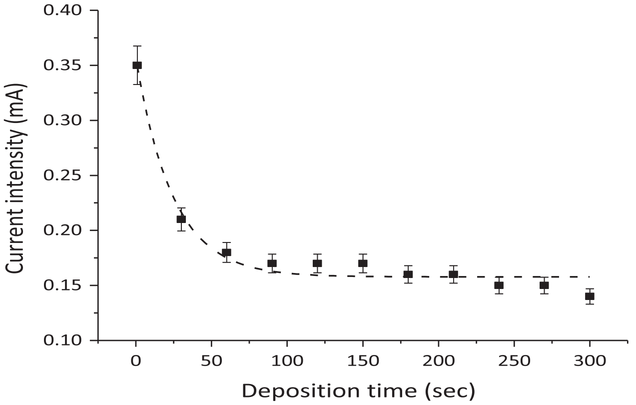

Fig. 4. EPD current intensity vs. deposition time for MUA-capped CdSe nanoparticles under an applied voltage of 5 V.

On the other hand, the washed MUA CdSe nanoparticles formed a uniform and strongly adhered film on the anode. Table 1 shows an increase in the amount of Cd and Se using particles washed once for EPD. This suggests that MUA molecules are loosely bound to the CdSe nanoparticles and are easily removed during washing. However, not all MUA molecules were washed away. If all MUA ligands on the CdSe particles were washed away, the CdSe nanoparticles would not be suspended in ethanol and would tend to sediment to the bottom of the vial. The removal of some MUA molecules would expose the Cd and Se sites at the surface. Table 1 shows that the number of Se sites exposed was greater than the number of Cd sites exposed, which possibly made the nanoparticles negatively charged and, under an applied voltage, caused them to drift toward the anode.

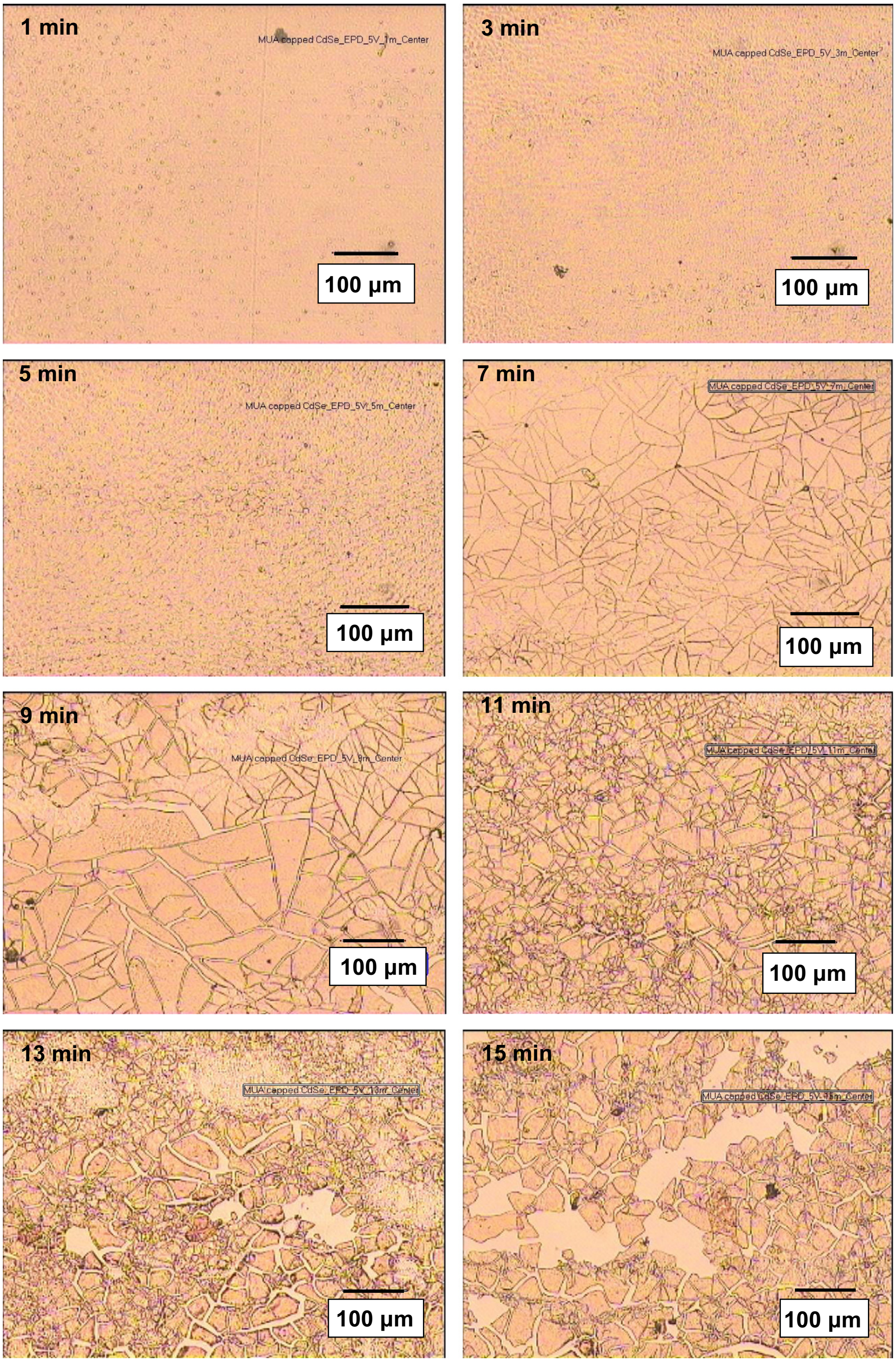

Fig. 5. Optical micrographs of MUA-capped CdSe nanoparticle films deposited at an applied voltage 5 V and deposition times of 1 to 15 min (magnification: 10X).

After washing the MUA-capped CdSe nanoparticles twice, some of the nanoparticles no longer dispersed uniformly in the suspension media and agglomerated at the bottom of the vial. This behaviour was possibly due to the complete loss of ligands during the washing procedure. Twice-washed nanoparticles showed no deposition on the substrate by electrophoretic deposition. It appears that the washing procedure reduces the density of the MUA ligands and hence reduces the repulsive force between particles. Under an applied potential, these particles tend to aggregate before they reach the electrode, which makes them sediment near anode.

3.2.4. *Deposition time of MUA-capped CdSe suspension*

The deposition time is also important to consider in electrophoretic deposition. In this experiment, MUA-capped CdSe nanoparticles with a diameter of 3.04 nm and a concentration of 3.67×10^{15} dots cm^{-3} were deposited by EPD. The voltage applied was 5 V, and the deposition times

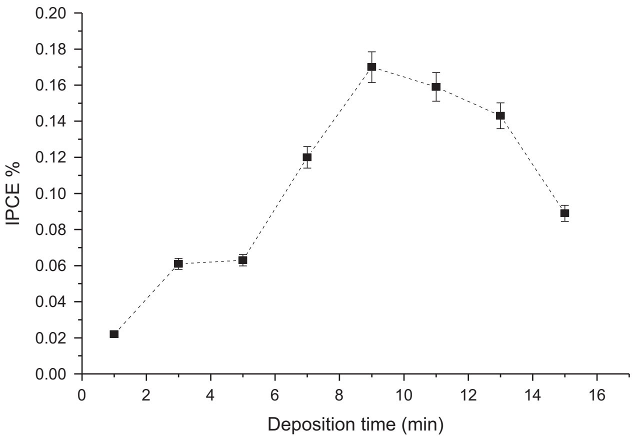

Fig. 6. Relationship between IPCE and thickness of MUA-capped CdSe nanoparticle films deposited at times ranging from 1 to 15 min.

studied were the range of 1 to 15 min. The film thicknesses were measured using a Zygo® White Light Interferometer at three different positions of the films. The average of three measurements with an associated error of 5% is reported in Fig. 3 for MUA-capped CdSe nanoparticles suspended in ethanol. During the first 5 min, the thickness increased dramatically with time, which led a high deposition rate and then levelled off after 5 min, which led to a low deposition rate. The decline of the deposition rate is directly associated with the decrease in current density shown in Fig. 4, which may be due to a drop in the potential across the film and therefore a lower electric field.

Fig. 5 displays optical micrographs of MUA-capped CdSe nanoparticles deposited at different times. The presented micrographs were taken at the centre of the deposited films after drying at room temperature. At the beginning of the deposition, no cracks were observed. Only at deposition times longer than 7 min did cracks start to form, which may have been caused by the evaporation of the solvent. The evaporation of solvent trapped in the interstices between the nanoparticles and the ligands during the drying process leaves nanovoids behind, which initiate the formation of cracks. As the deposition time increases, more cracks are generated due to the propagation of cracks along the nanovoids. If the crack length exceeds the critical crack length, islands form between cracks, which are susceptible to delamination [16]. This behaviour was observed at deposition times of 13 min, when the deposited film started to delaminate, and 15 min, when the delamination areas became larger.

3.3. Effect of deposition time of MUA-capped CdSe Nanoparticles on IPCE

In this experiment, the same samples used to study the deposition time were used. The samples were prepared from MUA-capped CdSe nanoparticles with a particle size of 3.04 nm and a concentration 3.67×10^{15} dots cm^{-3}. The voltage applied was 5 V, and deposition times in the range of 1–15 min were studied.

Photocurrent spectra were measured from a 1 mol dm^{-3} solution of Na_2SO_3 adjusted to pH 7. The light was chopped at a frequency 0.5 Hz, and a voltage of +0.4 V vs. Ag/AgCl/3 mol dm^{-3} KCl was applied to the test cells.

As shown in Fig. 6, as the deposition time increased to 5 min, i.e., as the film became thicker, the IPCE value increased. After 5 min of deposition, the thickness became more constant, but the IPCE value continued to increase up to 9 min of deposition and decreased thereafter.

The IPCE value increased during the first 5 min due to the increase in the amount of MUA-capped CdSe particles deposited on the substrate. For thin films, most of the photoexcited electron–holes generated were near the substrate, where electrons passed through a short pathway to transfer to the Fermi level of the substrate. For deposition times of 7 min and 9 min, the IPCE values increased, although the measured thickness remained approximately the same as that measured at 5 min of deposition. The increase in the IPCE may have been caused by the cracks generated in the films at deposition times of 7 min and 9 min (Fig. 5). The number of cracks enhanced the contact between the electrolyte and electrode surface, which also allowed for better photoexcited electron transfer to the substrate without electrons having to pass through the nanoparticles, making them more susceptible to recombination on their way to the substrate. At deposition times of 9 min to 15 min, the IPCE value decreased because the crack length increased and led to the delamination of the MUA-capped CdSe nanoparticle films, which resulted in fewer nanoparticles on the films to generate photoexcited electrons to transfer to the substrate.

4. Conclusions

The results of this study demonstrate that electrophoretic deposition can be successfully employed to deposit MUA-capped CdSe nanoparticles. The purification of MUA-capped CdSe nanoparticles is important in enabling the nanoparticles to move and adhere to the substrate during EPD. The apparent pH of the nanoparticles must be adjusted to pH > 10.3 to make them negatively charged. An applied voltage of 5 V was found to be suitable for EPD because at this voltage, no burning of the cathode occurred. A deposition time of 5 min yielded the highest-quality film with the fewest cracks. The thickness of the MUA-capped CdSe nanoparticle films increased exponentially at the beginning of deposition and levelled off after 5 min of deposition. Photocurrent spectroscopy carried out at 0.5 Hz in 1 mol dm^{-3} at pH 7 and an applied cell potential of +0.4 V vs. Ag/AgCl/3 M KCl. The resulting IPCE of 0.18% was the maximum value obtained in this study. This IPCE value demonstrates that CdSe quantum dot films deposited by electrophoretic deposition are suitable for use as photoanodes in quantum dot-sensitised solar cells.

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