The conversion of solar energy into chemical fuels through photocatalytic water splitting using semiconductors is considered one of the most promising ways to solve the energy crisis and global environmental issues. Since the first discovery of the photocatalytic splitting of water under ultraviolet light by Fujishima and Honda in 1972,1 many semiconducting materials and junctions have been studied as photoanodes for high efficiency photoelectrochemical (PEC) water-splitting systems for decades.2–4 Among them, n-type CdS is one of the attractive candidates due to its optimal bandgap (~2.4 eV), which makes it efficient for the absorption of visible light. In addition, the band position of the conduction band (CB) and valence band (VB) edge levels in the CdS semiconductor perfectly meets the driving condition for the redox reaction of water for photocatalytic water splitting, which makes it a suitable candidate.5,6 In the nano regime, it is well known that increase of the substrate temperature in either inert or ambient atmosphere leads to the increase of the grain size with the enhanced crystallinity. Therefore, post-heat-treatment of CdS films plays a vital role in the physicochemical, optical, and electrical properties of CdS.7 Ali et al. also reported about vacuum annealing to grow crystalline thin films for solar cell application.8

Recently, instead of using bare semiconductors as photocatalysts, several groups reported about enhanced photocatalytic activity of the material by precipitating metal particles on the interface of the semiconductor photocatalyst as catalysts. Gold nanoparticles (Au NPs) are preferred for introduction into semiconductor-based systems for visible-light-driven water splitting due to the generation of hot electrons by the unique localized surface plasmon resonance (LSPR) properties of gold.9–11 However, the average lifetime of hot electrons is very short (< 160 fs), and the injection rate of the hot electrons from Au NPs to CdS is relatively low; thus, the photocconversion efficiency is still unsatisfactory. Therefore, a material with high electron mobility that can produce electron transport is demanded. From this perspective, reduced graphene oxide (rGO) can be the good candidate. rGO has attracted significant attention for the wide variety of applications because of its two-dimensional honeycomb-like network with high specific surface area, excellent electrical conductivity, and good chemical and electrochemical stability. Furthermore, rGO reveals the property to form a heterojunction when incorporated with semiconductors.12–15 Jung et al. also demonstrated the formation of the metal sulfide and rGO heterojunction structure by spin coating of rGO.16

In this study, we rationally designed the CdS thin film via easy and low-cost chemical bath deposition (CBD), which was then annealed in an inert atmosphere at different elevated temperatures (400–600 °C) to produce a polycrystalline CdS phase with high crystallinity, followed by sequential spin-coating deposition of rGO and Au to form Au-rGO@CdS films. In such structure, the highly crystalline CdS thin film not only serves as an absorption layer but also serves as a high-speed transport path for the photogenerated electrons. The high conductivity rGO layer coated with Au NPs on CdS thin film structure was used as an electron sink to facilitate exciton separation17 and to store the separated electrons to increase the electron injection rate from Au NPs to CdS.17,18

Energy band diagram is suggested to elucidate the behavior of the photogenerated electrons and holes in detail. The PEC water splitting performance under visible light irradiation of the as-obtained photocatalysts is systematically investigated, and the enhancement mechanism of PEC activities in the CdS/rGO: Au is proposed.

Experimental

Fabrication of CdS thin films.—CdS thin films were prepared on FTO-coated glass substrates (10 Ω/□) via a modified chemical bath deposition (CBD) method, as shown in Fig. 1.5,9 2 mM cadmium nitrate tetrahydrate was complexed with 30% ammonia solution and then mixed with 10 mM thiourea as a sulfur source to form a precursor solution for the CdS deposition. The cleaned FTO substrates were immerged in the precursor solution and maintained at 70 °C for 3 h. This process was repeated up to five times for the same sample to thicken the thickness of the CdS thin film. The prepared samples were washed several times with deionized (D.I.) water and ethanol followed by drying at 70 °C. To improve the crystallinity, CdS nanoparticle thin films were annealed in an argon atmosphere at 500 °C for 1 h with a ramp rate of 5 °C/min. For the comparison, the annealing temperature was also set at 400 °C and 600 °C.

Coating of rGO and Au NPs.—rGO was coated onto the CdS thin film by spin-coating using graphene oxide prepared by the modified Hummers method.19 An optimized amount of graphene oxide (2 mg/ml) was dispersed by ultrasonication and mixed in a mixture of DI water and isopropanol (volume ratio 1:1) for 2 h at room temperature. The graphene oxide suspension was spin-coated for 15 s at 2000 rpm onto a CdS/FTO substrate 1, 2, 3, 4, and 5 times to achieve different thicknesses of rGO on the CdS/FTO substrates. The rGO-coated samples are denoted r(GO), where n represents the number of coating times. An approximately 8 μl solution of 2 mM HAuCl4 in isopropanol was subsequently spin-coated on the rGO@CdS/FTO film by spin-coating for 10 s at 2000 rpm. In addition, HAuCl4 solution was also coated on the CdS/FTO film to study the effect of rGO. Finally, the
Figure 1. Schematic flow for fabrication of Au-rGO@CdS thin film.

prepared photoanode structures were annealed at 320 °C for 2 h in an Ar atmosphere.\textsuperscript{21}

**Characterization of the thin film properties.**—Studies about the microstructure of the prepared samples were performed by field-emission scanning electron microscopy (FE-SEM, JSM700F, JEOL) and transmission electron microscopy (TEM, IEM-ARM200F, JEOL). Energy dispersive spectroscopy (EDS) equipped with TEM was used to study the compositional distribution in the structures. The microstructures and optical properties of samples were investigated by X-ray diffraction (XRD, Rigaku, D/MAX-RC) with Cu Ka radiation and UV-vis-NIR absorption spectroscopy, respectively. X-ray photoelectron spectrometry (XPS, Kratos Axis Ultra DLD) was used to analyze the surface composition of the samples. Raman spectroscopic analysis was performed using a UmiRam spectrometer with an excitation line of 532 nm. Photoluminescence (PL, LabRAM HR-80) characterization was employed to study the electron transport between CdS, rGO and Au.

**Measurement of photoelectrochemical cell performance.**—All PEC measurements were carried out by an electrochemical system (PGSTAT128N, Metrohm Autolab Instrument) with the three-electrode configuration using the prepared photoanode as the working electrode, a Pt plate as the counter electrode, and a saturated calomel electrode (SCE) as the reference electrode. The electrode area was fixed to have 0.5 cm\textsuperscript{2} with a nonconductive epoxy. An aqueous solution of 0.35 M Na\textsubscript{2}S and 0.25 M Na\textsubscript{2}SO\textsubscript{3} (pH ~ 13.5), where sulfuric phases serve as sacrificial agents, was used as the electrolyte. The photocurrent was measured under simulated sunlight illumination (1 Sun, 100 mW/cm\textsuperscript{2}) using a 150 W Xe arc lamp with an AM 1.5G filter. Prior to performing the experiment, the 1 Sun illumination condition was calibrated against a Si photodiode before PEC measurements. Photocurrent density-voltage characteristics (J-V curves) were obtained at a scan rate of 20 mV/s from the negative to positive potential direction. Electrochemical impedance spectroscopy (EIS) was conducted by applying an AC voltage amplitude of 10 mV with the frequency range from 0.01 to 100,000 Hz under AM 1.5 G illumination at −0.5 V vs SCE.

**Results and Discussion**

**Characterization of CdS thin film.**—The CdS sample which was prepared by four repeating CBD cycles shows the optimum growth condition of CdS thin films (Fig. S1). Figs. 2a–2d show FESEM images of the as-deposited CdS thin film and CdS thin films annealed at different temperatures. Along with the cross sectional image, the top view of all the samples is represented as an inset. As shown in Fig. 2a, the prepared CdS thin film with a thickness of ~300 nm was uniformly coated on the FTO substrates. From the cross sectional images, there is little difference in the thickness of the films. Meanwhile,

![Figure 2](image-url) Cross sectional FESEM images of CdS thin film annealed at different temperatures: (a) as-prepared, (b) 400°C, (c) 500°C, and (d) 600°C. The insets show top-view images.
on an absorption onset of hexagonal CdS structure with lattice parameters of \( a = 4.12 \) Å and \( c = 6.68 \) Å (JCPDS Card # 80-0006). The strong diffraction peak centered at the scattering angle of 26.6°, corresponding to the (002) plane of hexagonal CdS, which dominates the other peaks, evidences over the FTO substrate at the initial film growth stage. Diffraction peaks at 25.9°, 28.3°, 43.9° and 48.1° matching the (100), (101), (110) and (103) lattice planes, respectively, were prominent during the annealing process. This confirms the increase of crystallinity as well as the decrease in the defect densities in the CdS thin film during annealing process. No crystalline peaks from secondary phases, such as CdO, were observed, indicating that no phase conversion of CdS to CdO occurred during the annealing process. The as-prepared CdS thin film was found to be composed of hundreds of nanometer size nanochunks, which are then composed of tens of nanometer particles (nanopearls) without pinholes or voids. As the annealing temperature increases, compared to the as-deposited CdS thin film, the grain size of these nanopears increases due to the coalescence process leading to an increase of the crystallinity of the thin film. In the nano-regime, it is well known that as the substrate temperature decreases, the relatively low current density can be attributed to the smoothened surface morphology (inset of Fig. 2d), where less active surface area was effective to the absorption of light or charge transfer to water. 25 Meanwhile, relatively greater nanocrystals grown at 500 °C annealing produced a high crystallinity of the film. In addition, the vertically elongated CdS grains could be tightly adhere to the FTO substrate, which would result in lower resistance between the photoabsorption layer and the conductive substrate (Rs), enabling higher photovoltaic performance. Thus, the film annealed at 500 °C could be determined as a highly desirable microstructure for an efficient solar conversion device. Current densities and resistances of samples are tabulated in the Table I.

Characterization of the rGO@CdS thin film.—The surface morphology of the CdS thin film post-annealed at 500 °C followed by being spin-coated with rGO, rGO@CdS, is shown in Fig. 4a. After spin-coating and heat-treatment, a continuous and transparent layer of rGO formed on the surface of the CdS thin film. The existence of CdS and rGO was identified by the Raman spectrum (Fig. S4). The characteristic bands located at 307 and 605 cm\(^{-1}\) correspond to the fundamental optical phonon mode (1LO) and the overtone mode (2LO) of hexagonal CdS, respectively.26 Upon zooming in on the range of 1200–2400 cm\(^{-1}\), two typical peaks can be observed at 1347 and 1598 cm\(^{-1}\), which belong to the first-order D band and G band of rGO, respectively.29 The D band is related to the presence of defects and structural disorders in graphene sheets, while the G band is associated with the stretching of the sp\(^2\)-hybridized carbon-carbon bonds.27 Generally, the ratio of the intensity of the D band to the G band (I(D)/I(G)) is a measure of the degree of disorder in graphene films. The I(D)/I(G) intensity ratio is approximately 0.90, which is less than 1.

| Sample       | Current density at 0 V (vs SCE) | \( R_s \) (\( \Omega \)) | \( R_{\text{bulk}} \) (\( \Omega \)) | \( R_{\text{ct}} \) (\( \Omega \)) |
|--------------|--------------------------------|--------------------------|-------------------------------|---------------------------------|
| as prepared  | 1.4 mA/cm\(^2\)                | 63.5                     | 662.7                         | 2058                            |
| Cds-500      | 3.1 mA/cm\(^2\)                | 32.2                     | 250                           | 1573                            |
| rGO@Cds-500  | 3.4 mA/cm\(^2\)                | 33.3                     | 211                           | 1285                            |
| Au@Cds-500   | 3.8 mA/cm\(^2\)                | 45.5                     | 241                           | 187.2                           |
| Au-rGO@Cds-500 | 5 mA/cm\(^2\)               | 38.3                     | 204                           | 141.4                           |

Figure 3. (a) XRD patterns of the different CdS photoanodes: (1) as-prepared and (2), (3), and (4) CdS annealed at 400 °C, 500 °C, and 600 °C, respectively. (b) LSV curves obtained under dark and simulated sunlight illumination conditions of as-prepared CdS photoanodes and samples annealed at different temperatures.

Figure 4. (a) Top-view FESEM images of rGO@CdS. (b) LSV curves of rGO-modified CdS photoanodes with different amounts of rGO.
columnar grains, as shown in Fig. 6a, with excellent perpendicular photoelectrodes (Figs. 5a–5c). In the meantime, the CdS layer formed which were uniformly distributed on the surface of the rGO@CdS atoms were agglomerated to Au NPs with an average size of maximum value of 3.4 mA/cm² at 0 V (vs SCE) with a cycle so of CdS thin film, the photocurrent density showed little increase and the increase of the reaction rate. The flatband shift suggests that the band bending at the CdS surface in contact with the solution formed via the uniform distribution of Cd, S, and Au in the Au-rGO@CdS film.

Fig. 7a shows a survey scan of the XPS spectra for CdS and Au-rGO@CdS. The presence of elements such as Cd, S, C, O and Au was found through XPS. The high-resolution XPS spectrum in the Au 4f region, presented in Fig. 7b, shows the doublet peaks of Au 4f1/2 and Au 4f3/2 at approximately 83.6 and 87.3 eV, respectively. These characteristic peaks with a difference of 3.7 eV well matched with the metallic Au 4f state, indicating successful reduction of Au³⁺ to metallic Au on the surface of the electrode, rGO@CdS.²⁹

Figs. 7c and 7d shows the high-resolution XPS spectra of S 2p and Cd 3d, respectively, from the CdS and Au-rGO@CdS thin films. The CdS thin film post-annealed at 500°C showed energy binding at 405.1 and 411.8 eV for Cd 3d₅/₂ and Cd 3d₇/₂, respectively. The binding energies found at 161.1 and 162.4 eV indicated the presence of S 2p½ and S 2p¾, respectively. The peak positions of Cd 3d₅/₂ and S 2p showing core-level interactions matched with those of pure CdS.³⁰,³¹ For the Au-rGO@CdS, the aforementioned peaks of both Cd and S were shifted to lower binding energy. Binding energies of Cd 3d₅/₂ and Cd 3d₇/₂ shifted from 405.1 eV to 404.4 eV from 411.8 eV to 411.1 eV, respectively. S 2p½ and S 2p¾ also showed the negative shift from 161.1 eV to 160.8 eV and from 162.4 eV to 162.1 eV, respectively. The origin of the lower energy shift can be ascribed to the transfer of electron from Au particle to rGO@CdS, which has been reported as a common phenomenon in semiconductor Au heterojunctions.³¹–³² Furthermore, high-resolution spectra in the region of the C 1s and O 1s characteristic peaks for the Au-rGO@CdS thin film are shown in Figs. 7e and 7f. The deconvolution of peaks shows the presence of rGO indicated by the emergence of the peak at 284.6 eV as the major feature of the C 1s, which can be assigned to the C−C and C=O bonds corresponding to the sp² carbon. Oxygen-containing carbonaceous bonds were also detected at higher binding energy levels, such as 286.3, 287.7, and 289.5 eV associated with C−OH, C=O, and −COOH groups, respectively.³³

Simultaneous integration of rGO and Au NPs, Au-rGO@CdS, resulted in the highest photocurrent density of 5.0 mA/cm² at 0 V vs SCE (Fig. 8a). The effect of Au on the photocurrent enhancement is attributed to the plasmonic effect of Au NPs, which is beneficial for light absorption of the structure, as will be discussed later along with Fig. S5. The photocurrent density of 3.8 mA cm⁻² from the Au@CdS photoanode was much lower than that from the Au-rGO@CdS, suggesting that rGO can serve as an electron mediator and charge transport layer between Au NPs and CdS.

![Figure 6.](image-url) (a) TEM image and (b-d) EDS elemental mapping of Au-rGO@CdS for Cd, S, and Au.

![Figure 5.](image-url) (a) Top-view FESEM image, (b-c) HRTEM cross-section images of Au-rGO@CdS.
This assumption can be corroborated by the electrochemical impedance spectroscopy (EIS) analysis shown in Fig. 8b, which again indicates that all photoelectrodes exhibited Randles circuit behavior. The EIS analysis distinguished the contribution of each photoanode component to enhancement of the photocurrent density. The variables $R_s$, $R_{\text{bulk}}$, and $R_{\text{ct}}$ can be termed by the series resistance at the interface between the FTO substrate and thin film, the surface states charge trapping resistance, and the charge transfer resistance, respectively, with their fitting values listed in Table I. The smallest $R_{\text{ct}}$ value for Au-rGO@CdS-500 was observed, which indicates that $R_{\text{ct}}$ dramatically decreased after loading of Au and rGO. However, it should be noted that decrease of $R_{\text{ct}}$ by Au is far greater than that by rGO. The $R_{\text{bulk}}$ for Au-rGO@CdS-500, 204 $\Omega$, was found to be smaller than that of the other photoanodes. This results from the least recombination of the electrons and holes at the interface of the photoelectrode/electrolyte, possibly leading to the high current density in the system. Meanwhile, it is also confirmed that the effect of Au on CdS results in the slight decrease of $R_{\text{bulk}}$ from 250 $\Omega$ to 241 $\Omega$ compared with CdS, which is not sufficient to lower the recombination rate.

The charge separation could be directly investigated by photoluminescence (PL) spectra. For semiconductor nanomaterials, the PL spectra are related to the transfer behavior of the photoinduced electrons and holes, which reflects the separation and recombination of photoinduced charge carriers. Particularly, the electron–hole pairs recombine to release the extra energy via light emission. If the electron–hole pairs are well separated, the possibility for them to recombine decreases, leading to so-called PL quenching. Fig. S5 shows the PL spectra of the CdS and those with rGO and/or Au. The CdS thin film spectrum exhibiting a strong emission peak centered at approximately 512 nm revealed a bandgap of $\sim$2.4 eV for CdS. The PL intensities decreased with the addition of rGO, which can be understood as a strong absorption in rGO and consequently reduced absorption in CdS. However,
the apparent redshift of the peak by Au NPs to $\sim$560 nm in Au/CdS is originated from the surface plasmonic effect of Au, with fully suppressed absorption in CdS. For the Au/rGO/CdS, the peak intensity further decreased along with the absorption in the rGO.

The photocurrent density vs potential (vs SCE) curves for different photoelectrode structures, Fig. 8a, can be understood using the band diagrams (Fig. 9). Fig. 8a reveals that the flatband potential ($V_{fb}$), which was measured with respect to the reference electrode SCE in the three-electrode configuration, was $-1.32$, $-1.47$, and $-1.42$ V for CdS, rGO@CdS, and Au-rGO@CdS, respectively. The Mott-Schottky measurements in dark condition exhibited similar turn-on potentials among the samples as illustrated in Fig. S6. The negative $V_{fb}$ indicates that the band of the electrode is bent up at the contact with the electrolyte. The energy level of the SCE in the electrolyte is independent of the pH of the solution and is given by $E_{SCE} = E_{vac} - E_{HER} = -4.74$ eV since $E_{vac} - E_{HER} = -4.5$ eV, where $E_{HER}$ is the energy of the hydrogen evolution reaction. On the other hand, the Fermi level of the solution depends on the pH and is given by $\phi_{aq} = -4.5 - \frac{12}{13} + 0.059 \times 13.5 = -4.32$ eV. The energy levels are shown in Fig. 8a. For the working electrode, the electron affinity of CdS ($\chi_{CdS}$) was taken as 3.6 eV, assuming a Fermi level of 0.2 eV below the conduction band edge, as shown in Fig. 9a.

At the thermal equilibrium of CdS in the electrolyte, the Fermi energies should be aligned. Band bending (or built-in potential $U_b$) forms as $U_b = E_{F,CdS} - E_{F,aq}$, which is different from the $V_{fb}$.

![Figure 8](image-url)  
**Figure 8.** (a) LSV curves and (b) Nyquist plot of various photoelectrodes under simulated sunlight illumination.

![Figure 9](image-url)  
**Figure 9.** Schematic for the energy bandgap diagrams of Au@CdS and Au-rGO@CdS before and after contact with each other.
measured with respect to the reference electrode, or $E_{F,Cds} - E_{SCE}$. According to this relationship, the band bending at the CdS surface is $1.32 - (4.74 - 4.32) = 0.90$ eV, as sketched in Fig. 9b. It should be noted that the band bending calculated from $E_{F,Cds} - E_{F,aq}$ in Fig. 8a is $0.52$ eV ($= 4.32 - 3.8$), which is the band bending without any Helmholtz layer induced by the pH of the solution. Therefore, the difference in the band bending between the measured value and what was estimated by the band diagram parameters (0.90-0.52 = 0.38 eV) was attributed to the potential drop at the interface by the Helmholtz layer. In this way, the measurements and the material parameters could be correlated to show the band diagram of the CdS/(0.35 M Na$_2$S + 0.25 M Na$_2$SO$_3$ electrolyte) PEC system in Fig. 9b.

For the rGO/CdS system, the greater $V_{fb} = -1.47$ V led to a built-in potential of 1.05 V resulting from the higher Helmholtz potential, as shown in Fig. 8c. The work function for rGO ($\phi_{rGO}$) was taken as 4.7 eV. The increased band bending with rGO can be explained by the formation of an rGO/CdS p-n junction. However, the Au decoration on CdS resulted in a reduced $V_{fb}$ of $-1.14$ eV (Fig. 8a) following by a decreased band bending at the interface. For a continuous Au layer with a work function of 5.3 eV, $V_{fb}$ greatly increased band bending of $-1.5$ eV is attributed to the formation of Schottky junction, which corresponds to $V_{fb} = -1.92$ V. The much smaller band bending with Au decoration could be observed because Au served as a catalytic nanoparticle rather than a film. The coating of Au particles on rGO (or the sample Au-rGO@CdS) also showed reduced band bending, shown in Fig. 9d as estimated from $V_{fb} = -1.42$ eV. The increase of the photocurrent by rGO itself is not noticeable. However, the Au decoration effect on the photocurrent enhancement was great and stimulated by the surface plasmonic resonance effect that enhanced the injection of electrons into CdS as well as holes into the electrolyte. The rGO effect on the increase of the current is not direct but definitely beneficial to the uniform distribution of Au particles, leading to the enhanced SPR effect.

Conclusions

We reported the development of the photo-efficient Au-rGO@CdS photoelectrode using simple spin-coating of rGO and Au NPs on the surface of a CdS thin film. Along with the reduction of grain boundaries in the CdS thin film during the annealing process, the enhancement in the photo-generated charge conduction could be found in the composite photoanode. In detail, the optimal structure of the CdS thin film post-annealed at 500 °C showed the highest photocurrent density of 3.1 mA/cm$^2$ at 0 V (vs SCE). The Au-rGO@CdS revealed a further improvement in PEC activity, showing 5.0 mA/cm$^2$ at 0 V (vs SCE), which is an approximately 3.5 times photocurrent density increase compared to the as-prepared CdS thin film reference. The PEC enhancement with the Au-rGO@CdS structure can be understood due to the LSPR of Au NPs, which significantly improved the efficiency of photon capture while rGO effectively distributed Au particles on the surface. We scrutinized the energy diagram of the cell components, thus providing better understanding of the enhancement mechanism of PEC cells. In-depth study about various semiconductor/metal junctions is currently underway to further improve the PEC performance.

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