

Synergistically enhanced ternary Schottky/S-scheme homojunction for ultrasensitive photoelectrochemical sensing

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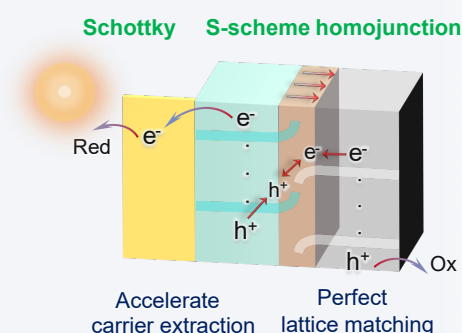
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ABSTRACT: Currently, constructing heterojunctions to improve photoelectric conversion efficiency represents a highly promising approach for improving the sensitivity in photoelectrochemical (PEC) sensors. Nevertheless, the performance enhancement is often constrained by the lattice mismatch between dissimilar semiconductors. In this work, we introduce a novel synergistic enhancement model based on a Schottky-coupled S-scheme homojunction. By controlling different conditions under the same hydrothermal method, the homojunction of CdS quantum dots grown *in-situ* on CdS nanorods was synthesized. The homojunction features intimate interfacial contact, a perfectly matched lattice, and an S-scheme carrier pathway, thus increasing the photocurrent response by 17 times. Subsequent integration with gold nanoparticles further amplifies the performance, yielding a synergistic 31-fold improvement. Leveraging this architecture, we demonstrate an ultrasensitive PEC biosensor for the detection of C-reactive protein activity, operating at 0 V bias and without sacrificial agents. This pioneering strategy of combining Schottky junctions with S-scheme homojunctions offers a new direction for advancing photoelectric performance in PEC sensing.

KEYWORDS: S-scheme homojunctions, Schottky junctions, lattice matching, C-reactive protein, photoelectrochemical sensing



1 Introduction

Photoelectrochemical (PEC) analysis is currently regarded as a considerably promising technique for ultrasensitive detection [1–4]. However, the photoelectric conversion efficiency of single semiconductors is generally low, leading to insufficient sensitivity of PEC sensors, which limits their further development [5, 6]. Currently, most PEC sensors rely on the construction of heterojunctions to achieve signal amplification, owing to the feasibility and effectiveness of heterostructures in spatially separating electron-hole pairs [7, 8]. Among various reported heterojunctions, advanced S-scheme heterojunctions have garnered significant attention due to their strong interface driving forces and redox capabilities [9–12]. In particular, when heterojunctions are

constructed via *in-situ* growth methods, the intimate contact further enhances interfacial carrier migration [13]. Despite this, lattice mismatch caused by differences in elemental composition and inconsistent interplanar spacing in heterojunctions can lead to reduced carrier mobility and increased recombination rates at the interface, which seriously hinders the optimization of the photoelectric properties of photosensitive materials [14]. Consequently, in previous PEC sensing studies, it has been necessary to apply a bias potential and introduce scavenging agents to accelerate carrier transport and extraction, thereby improving detection sensitivity [15]. However, the applied bias potential may introduce undesirable background noise and result in signal distortion, while relatively high voltages may also damage the electrode surface. Moreover, the introduction of scavenging agents limits the practical application scenarios of PEC sensors in complex environments.

In contrast to conventional heterojunctions, homojunctions constructed from semiconductors with identical elemental compositions exhibit lower interfacial defects, which can significantly suppress non-radiative carrier recombination [16–19]. However, common strategies for building homojunctions include element doping, dopant incorporation, and defect engineering

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[20–23]. These strategies still lead to lattice defects and fail to thoroughly address interface mismatch. Notably, from the perspective of microscopic interfacial contact, a smaller difference in lattice spacing corresponds to reduced interfacial stress and a lower density of defect states. While optimizing the homojunction interface contact, constructing an efficient S-scheme charge migration pathway is also critical [24, 25]. Therefore, rational design of lattice-perfect S-scheme homojunctions to significantly optimize photoelectric performance remains a major challenge for the development of ultrasensitive PEC sensors under 0 V bias and without the use of scavenging agents.

In this work, we report a lattice-matched CdS homojunction (CdS_{QR}) with an S-scheme charge transfer pathway, which is constructed through *in-situ* growth of CdS quantum dots (QDs) on CdS nanorods (NRs). Experimental investigations confirm highly consistent crystalline structures and lattice fringes between the two components, attributable to their identical chemical composition and synthetic pathway. The optimal homojunction (with 40 wt.% QDs loading) exhibits a 17-fold enhancement in PEC performance over pristine CdS NRs. Further modification with Au nanoparticles (Au NPs) creates a Schottky-coupled S-scheme homojunction ($\text{Au-CdS}_{\text{QR}}$) architecture (Scheme 1) [26, 27]. It was found that the applied bias photon-to-current efficiency increased from 1.03% at 0.56 V for the unmodified control to 3.75% at 0.23 V. As a result, this hybrid configuration exhibits a synergistic effect, resulting in a 31-fold enhancement. Subsequently, the C-reactive protein (CRP)-specific aptamer was immobilized on the electrode surface via Au–N bonds, enabling high-affinity and selective recognition of the target protein. Leveraging the observed signal-on photocurrent response induced by low concentrations of CRP, we achieved ultrasensitive detection under 0 V bias and without scavenging agents, and obtained a limit of detection (LOD) as low as 5 pg/mL. This work demonstrates the great potential of Schottky-coupled lattice-matched S-scheme homojunctions in achieving ultrasensitive PEC biosensing.

2 Experimental

2.1 Synthesis of CdS NRs

Briefly, 2.3 g of $\text{CdCl}_2 \cdot 2.5\text{H}_2\text{O}$ and 4.5 g of NH_2CSNH_2 were dissolved in 60 mL of $\text{C}_2\text{H}_5\text{N}_2$. The mixture was subjected to 30 min of ultrasonic treatment, then transferred to a 100 mL polytetrafluoroethylene-lined reactor and allowed it to react at 160 °C for 48 h. After cooling to room temperature, the yellow precipitate (CdS NRs) was collected by centrifugation. The precipitate was washed three times alternately with deionized water and ethanol, and the final product was obtained by freeze-drying.

2.2 Synthesis of CdS quantum dots-decorated CdS nanorods (CdS_{QR})

To investigate the influence of QD loading, samples with theoretical CdS QDs mass fractions of 5%, 10%, 20%, 40%, and 60% relative to the CdS NRs support were prepared. In a typical procedure, 0.25 g of CdS NRs was dispersed in 70 mL of ethanol and ultrasonicated for 0.5 h to form a homogeneous suspension. Under continuous vigorous magnetic stirring, a corresponding stoichiometric ratio of $\text{Cd}(\text{AcO})_2 \cdot 2\text{H}_2\text{O}$ was added, followed by an equimolar amount of NH_2CSNH_2 . The resulting mixture was transferred into a Teflon-lined autoclave and reacted at 120 °C for 10 h. After the reaction, the yellow precipitate (CdS_{QR}) was collected by centrifugation (6000 rpm, 15 min), washed three times with deionized water and anhydrous ethanol, and finally obtained by freeze-drying.

2.3 Preparation of gold-coupled CdS homojunction ($\text{Au-CdS}_{\text{QR}}$)

Firstly, 10 μL of the as-prepared CdS_{QR} dispersion (5 mg/mL) was coated onto the surface of a pre-treated ITO electrode, followed by drying at 50 °C to obtain the CdS_{QR} -modified electrode. Subsequently, 10 μL of an Au NPs solution was carefully deposited onto the electrode surface after drying at 50 °C. The synthesis procedure for Au-CdS NRs was identical to that of $\text{Au-CdS}_{\text{QR}}$, except that CdS_{QR} was replaced by CdS NRs.

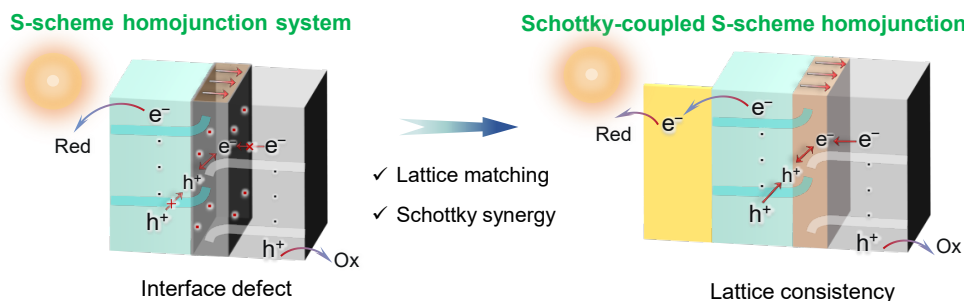
2.4 PEC sensing of CRP

The CRP detection protocol comprised three sequential functionalization steps: aptamer immobilization: A 10 μL aliquot of aptamer solution (2 μM) was drop-coated onto the pre-fabricated ITO electrode and incubated overnight at 4 °C to facilitate thiol-gold binding. Unbound aptamers were removed by rinsing three times with PBS (pH 7.2–7.4), followed by nitrogen drying. Surface passivation: To block non-specific binding sites, 10 μL of 1-hexanethiol (0.1 mM) was applied to the aptamer-functionalized electrode surface and incubated for 20 min at 37 °C. Subsequent PBS washing eliminated excess reagents, minimizing background interference. Target capture: 10 μL of CRP (0, 0.025, 0.05, 0.10, 0.20, 0.50, 1.0 ng/mL) solutions at graded concentrations were introduced onto the modified electrode and incubated for 30 min at 37 °C, enabling specific aptamer-CRP complex formation. Unbound CRP was removed via PBS rinsing.

3 Results and discussion

3.1 Design and characterizations of the materials

As a proof of concept, the $\text{Au-CdS}_{\text{QR}}$ is fabricated as a desired platform for the investigation of PEC biosensors. The detailed



Scheme 1 Schematic and the concept of designing Schottky-coupled S-scheme homojunction.

preparation process is manifested in Fig. 1(a). Firstly, CdS NRs are synthesized via hydrothermal synthesis. CdS_{QR} homojunctions are prepared by further using the hydrothermal method to *in-situ* grow CdS QDs on CdS NRs.

Transmission electron microscopy (TEM) and scanning electron microscopy (SEM) exhibit the successful preparation of CdS NRs (Figs. S1 and S2 in the ESM). As shown in Fig. S3 in the ESM, a high-resolution TEM (HRTEM) image reveals that the lattice spacing in CdS NRs is 0.248 and 0.331 nm, corresponding to the (102) and (002) planes, respectively. The CdS_{QR} retained the nanorod structure (Fig. 1(b)). HRTEM imaging of CdS_{QR} clearly shows that CdS QDs are grown *in-situ* on the CdS NRs, with a lattice spacing of 0.248 nm corresponding to the (102) plane, which aligns perfectly with that of the CdS NRs (Fig. 1(c)). X-ray diffraction (XRD) patterns verify the successful preparation of CdS NRs, with characteristic peaks of CdS (JCPDS No. 77-2306) [28]. The XRD pattern of CdS_{QR} matches well with that of the CdS NRs, confirming their identical crystal structure and providing direct evidence for the well-matched lattice interface in the homojunction (Fig. 1(d)). As evidenced by Fig. S4 in the ESM, TEM shows Au NPs with a size of about 20 nm. No distinct diffraction peaks of Au NPs are observed in the XRD pattern of Au-CdS_{QR}, which can be attributed to the low loading amount of Au. The presence of corresponding elements is further demonstrated by X-ray photoelectron spectroscopy (XPS) analysis (Fig. S5 in the ESM). These results collectively confirm the successful fabrication of Au-CdS_{QR} and the perfectly matched lattice interface within the homojunction.

3.2 Photoelectric properties

The photoelectric performance is subsequently characterized. The optimal photocurrent of CdS_{QR} is achieved when the loading of CdS QDs reached 40 wt.% (Fig. S6 in the ESM). Unless otherwise specified, all subsequent references to CdS_{QR} correspond to the

homojunction with this 40 wt.% CdS QDs loading. Following the trend in Fig. 2(a), the photocurrent of CdS NRs, CdS_{QR}, and Au-CdS_{QR} increased successively, indicating a gradual improvement in carrier separation efficiency. Notably, the Au-CdS_{QR} exhibits a photocurrent of 8.5 μ A, which represents a 31-fold enhancement compared to the pristine CdS NRs. This performance demonstrates a synergistic enhancement effect, as it significantly exceeds the improvements observed for CdS_{QR} (4.6 μ A, 17-fold enhancement over CdS NRs) and Au-CdS NRs (0.4 μ A, 1.5-fold enhancement over CdS NRs). The Nyquist plots are simulated by the equivalent circuit to investigate the charge transfer resistance (R_{ct}) in Fig. S7 and Table S1 in the ESM [29]. The R_{ct} progressively decreased from 19.2 Ω for CdS NRs to 16.5 Ω for CdS_{QR} and further to 14.8 Ω for Au-CdS_{QR}, suggesting a corresponding acceleration of charge carrier migration efficiency across the materials. The current density–voltage (J – V) curves of CdS NRs, CdS_{QR}, and Au-CdS_{QR} are measured (Fig. 2(b)), revealing a monotonic enhancement in performance that is consistent with the photoelectrochemical measurements. The applied bias photon-to-current efficiency (ABPE) plot (Fig. 2(c)) reveals a maximum ABPE of 3.75% for Au-CdS_{QR}, which is 3.6 times higher than that of CdS NRs (1.03%), demonstrating the superior photoelectric conversion efficiency of Au-CdS_{QR} [30]. Steady-state photoluminescence (PL) spectra exhibit significant quenching in Au-CdS_{QR}, suggesting more effective suppression of radiative recombination and enhanced charge carrier separation (Fig. S8 in the ESM) [31, 32].

3.3 Charge transfer mechanism

In-situ Kelvin probe force microscopy (KPFM) is further employed to visualize the surface potential distribution and corroborate the remarkable enhancement in photoelectric response [33, 34]. As presented in Fig. 2(d) and Fig. S9 in the ESM, the CdS_{QR} exhibits a more pronounced three-dimensional (3D) surface potential distribution (37 mV) in darkness compared to CdS NRs (11 mV),

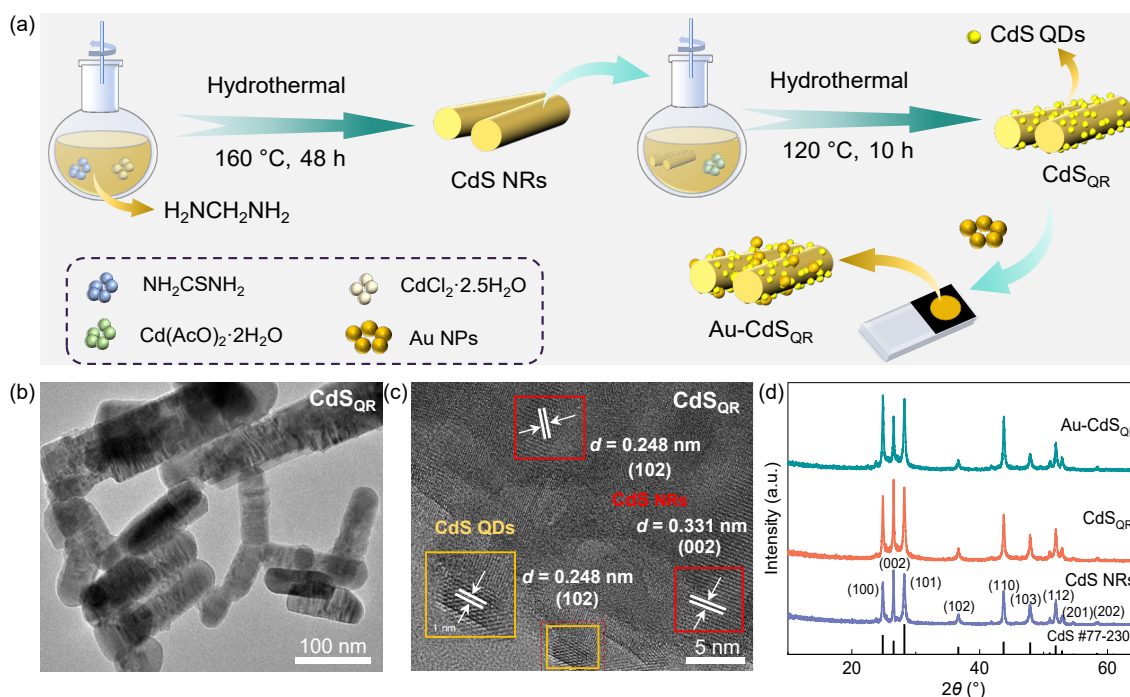


Figure 1 (a) Schematic illustration of the synthetic procedure for Au-CdS_{QR}. (b) TEM and (c) HRTEM images of CdS_{QR}. (d) XRD patterns of CdS NRs, CdS_{QR}, and Au-CdS_{QR}.

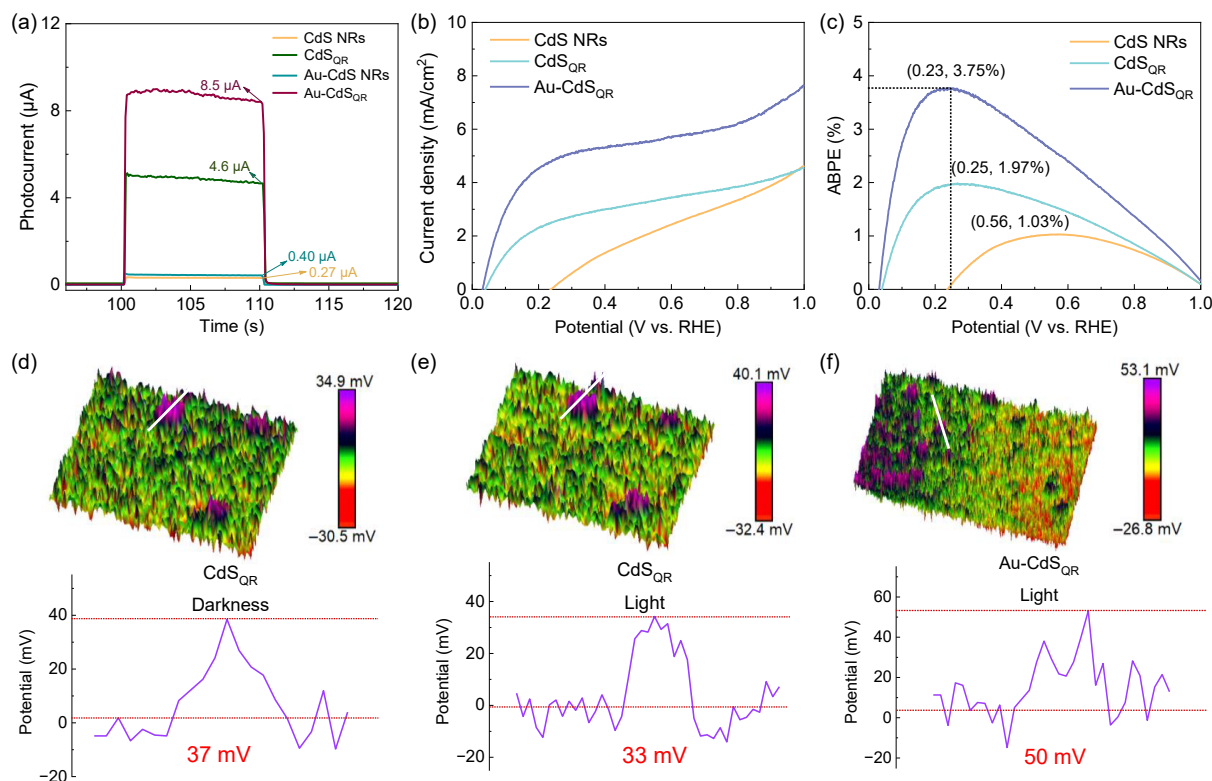


Figure 2 (a) Comparative photoelectric properties of CdS NRs, CdS_{QR}, Au-CdS NRs, and Au-CdS_{QR}. (b) *J*-*V* curves and (c) ABPE curves of CdS NRs, CdS_{QR}, and Au-CdS_{QR}. 3D surface potential distribution and corresponding line-scanning of (d) CdS_{QR} in darkness, (e) CdS_{QR} and (f) Au-CdS_{QR} under illumination detected with KPFM.

indicating a stronger built-in electric field within the homojunction that promotes the interfacial migration of photogenerated carriers. Moreover, the corrected potential distribution confirms that electrons transfer from the surface CdS QDs to the CdS NRs during homojunction formation. Under light irradiation, the surface potential of the CdS_{QR} is significantly reduced by 4 mV compared with that under dark conditions, indicating that photogenerated electrons accumulate on the CdS QDs surface (Fig. 2(e)). This behavior supports an S-scheme charge transfer mechanism, where photogenerated electrons move from the CdS NRs to the CdS QDs driven by the built-in electric field [35]. For Au-CdS_{QR}, the surface potential under illumination reaches 50 mV, indicating that the incorporation of Au NPs further accelerates the extraction of photogenerated carriers, thereby enhancing the photoelectric response (Fig. 2(f)).

Subsequently, band structure characterization and *in-situ* irradiated XPS analysis are employed to investigate the charge transfer pathways in CdS_{QR} and Au-CdS_{QR}. From the Tauc plots of UV-vis diffuse reflection spectral conversion, the band gaps (E_g) of CdS NRs, CdS_{QR}, and Au-CdS_{QR} are 2.37, 2.39, and 2.36 eV, respectively (Fig. 3(a) and Fig. S10 in the ESM). The similar absorption profiles demonstrate their comparable light-harvesting capabilities. As illustrated in Fig. S11 in the ESM, the positive slope of the Mott-Schottky curve tangent of CdS NRs, CdS_{QR}, and Au-CdS_{QR} indicates their n-type behavior. Therefore, the conduction band (CB) edge of CdS NRs, CdS_{QR}, and Au-CdS_{QR} are -0.50, -0.56, and -0.51 V (vs. NHE, pH = 0). The valence band (VB) potentials are 1.87, 1.83, and 1.85 V (vs. NHE, pH = 0), respectively. In combination with XPS VB spectra, the Fermi level (E_F) of CdS NRs, CdS_{QR}, and Au-CdS_{QR} can be calculated as 0.67, 0.45, and 0.35 eV (vs. NHE, pH = 0) (Fig. S12 in the ESM) [36]. Compared to CdS

NRs, CdS_{QR} exhibits lower energy positions of the CB, VB, and E_F , indicating that the CdS QDs possess lower corresponding energy levels than the CdS NRs. Furthermore, Au-CdS_{QR} shows more positive CB and VB energies but a lower E_F relative to CdS_{QR}, suggesting that the E_F of the Au NPs lies below that of CdS_{QR}. As illustrated in Figs. 3(b) and 3(c), after the *in-situ* growth of CdS QDs, the binding energies of Cd 3d and S 2p for CdS_{QR} exhibit a positive shift in the dark compared to CdS NRs. Since XPS probes chemical information from the extreme surface region, these shifts indicate electron transfer from the surface CdS QDs to the underlying CdS NRs in darkness. After light irradiation, the negative movement of Cd 3d and S 2p peaks indicates electron transfer from CdS NRs back to CdS quantum dots, which aligns consistently with the KPFM results. Similarly, compared with CdS_{QR}, the binding energies of Cd 3d and S 2p for the Au-CdS_{QR} negatively shift in darkness and positively shift under light irradiation, revealing the transfer of electrons from AuNPs to CdS_{QR} in darkness and the opposite transfer under illumination (Figs. 3(d) and 3(e)). The binding energies of Au 4f for the CdS_{QR} negatively shift under light irradiation, indicating electron transfer from CdS_{QR} to AuNPs (Fig. 3(f)). This indicates that the E_F of Au is lower than that of CdS_{QR}, which is consistent with the results derived from the band structure characterization. Based on these results, the band structure and electron transfer paths of CdS_{QR} and Au-CdS_{QR} in the PEC process are displayed in Fig. 4. It can be seen that the CdS homojunction follows an S-scheme charge transfer mechanism, while the integration of Au NPs forms a Schottky junction that further accelerates charge carrier migration.

3.4 Performance of the PEC biosensor for CRP detection

As a proof of concept, we used the Schottky-coupled S-scheme

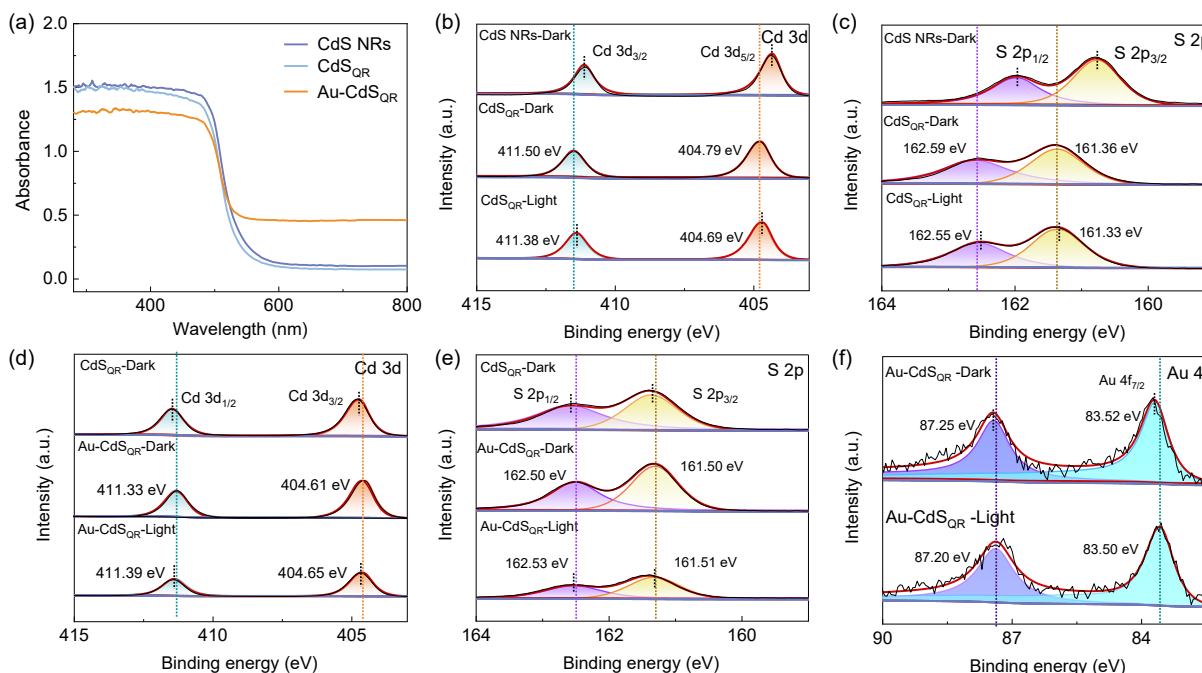


Figure 3 (a) UV-vis diffuse reflectance spectra. High-resolution XPS spectra of (b) Cd 3d and (c) S 2p for CdS NRs and CdS_{QR}. High-resolution XPS spectra of (d) Cd 3d, (e) S 2p, and (f) Au 4f for CdS_{QR} and Au-CdS_{QR} in darkness and under 365 nm illumination.

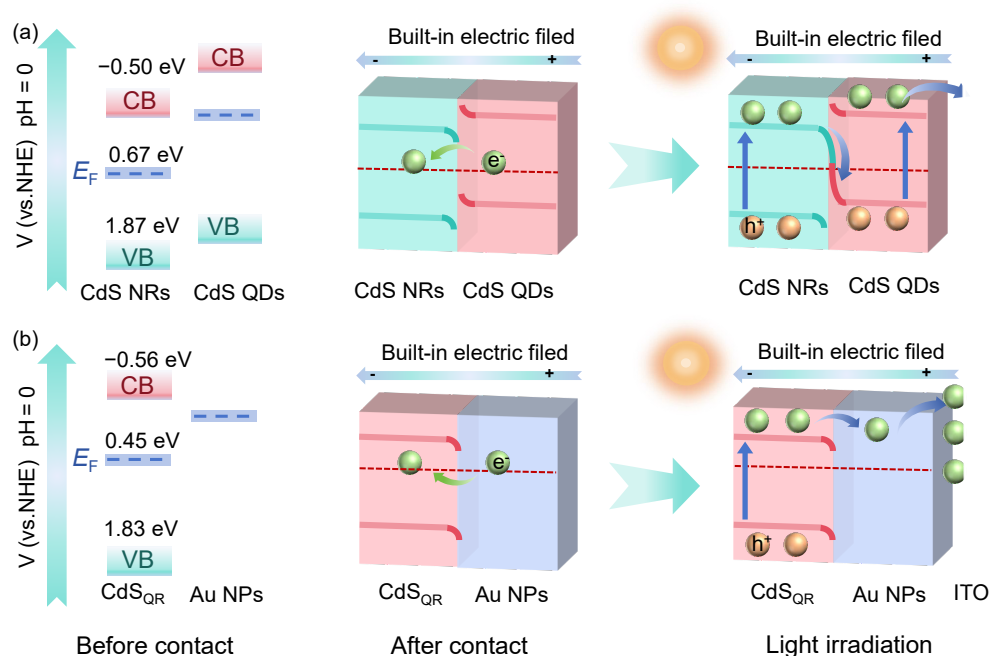


Figure 4 Band structure and charge transfer path schematic diagrams of (a) CdS_{QR} and (b) Au-CdS_{QR}.

homojunction in a PEC biosensor to detect CRP, a sensitive inflammatory biomarker (Fig. 5(a)). High-sensitivity CRP independently predicts cardiovascular events and has critical clinical value for infection, inflammation, and cardiovascular risk assessment. PEC sensing features inherent separation of excitation and detection signals, offering low background noise, rapid response, and label-free operation, thus providing an ideal platform for the ultrasensitive and point-of-care detection of CRP. The CRP-specific aptamer, modified with an amino group, was immobilized via Au-N bonds. After blocking nonspecific adsorption sites with 1-hexanethiol (HT), different concentrations of CRP were specifically

captured by the aptamer. The variation in photocurrent response was achieved through the modulation of electron transfer at the solid-liquid interface by CRP, enabling quantitative detection of CRP. The results of the incubation time optimization showed that the signal remained stable after 30 min. Therefore, 30 min was selected as the optimal incubation time (Fig. S13 in the ESM). The detection feasibility was also verified (Fig. S14 in the ESM). As presented in Fig. 5(b), under the conditions of 0 V bias and without scavenging agents, the photocurrent response initially increased and then decreased with increasing CRP concentrations. This trend may be attributed to the reaction between CRP and holes at lower

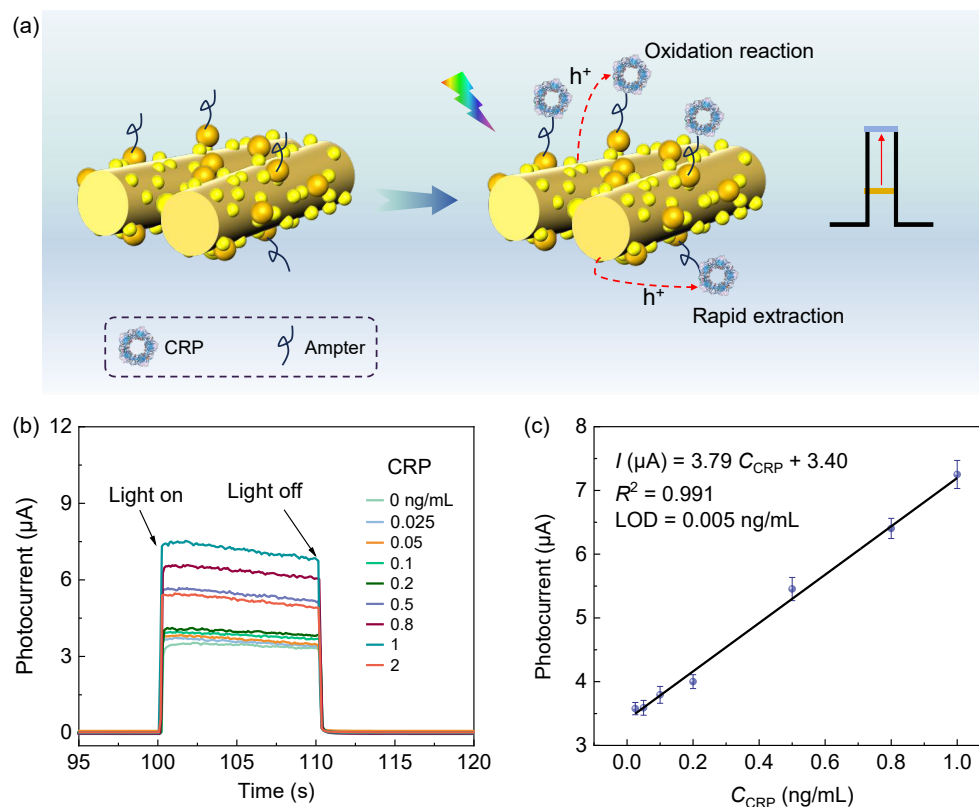


Figure 5 (a) Mechanism diagram of CRP sensing detection. (b) Current–time (*i*–*t*) curves at different concentrations of CRP detection (0–2 ng/mL). (c) Linear calibration curve of the PEC biosensor in the range of 0.025–1 ng/mL (error bars are based on the standard deviation of the mean, *n* = 3).

concentrations and the steric hindrance effect at higher concentrations. Electrochemical impedance spectroscopy (EIS) also displays the feasibility of the detection of CRP and the corresponding R_{ct} changes (Fig. S15 and Table S2 in the ESM). The photocurrent is linearly correlated with CRP within the scope of 0.025–1 ng/mL (Fig. 5(c)). The linear fitting equation is $I (\mu A) = 3.40 + 3.79 C_{CRP}$ ($R^2 = 0.991$), and the LOD (S/N = 3) is 5 pg/mL. In addition, the prepared PEC biosensor manifests excellent selectivity, anti-interference capability, and reproducibility (Figs. S16–S18 in the ESM). Notably, when benchmarked against existing approaches (Table S3 in the ESM), the present PEC biosensor exhibits highly competitive sensitivity. Detection of actual human serum samples showed that the CRP concentrations of five healthy individuals were all within the normal range, which confirms the reliability of this biosensor (Table S4 in the ESM). The above results underscore the utility of the developed PEC biosensor, indicating its wide applicability in the realms of clinical diagnosis and biomedical research.

4 Conclusions

In summary, we have demonstrated an innovative model of a novel Schottky-coupled S-scheme homojunction (Au–CdS_{QR}). The variation of controlled conditions within the same hydrothermal method provides an effective reference for fabricating lattice-perfect S-scheme homojunctions. Further integration with Au nanoparticles to construct a Schottky junction exhibits synergistic enhancement of the photocurrent characteristics, ultimately achieving a remarkable 31-fold improvement. Based on this advanced photoactive material, we have developed an ultrasensitive photoelectrochemical biosensor for detecting CRP that operates

at 0 V bias and without sacrificial agents. This paradigm offers a universal pathway for designing efficient photoelectric materials for next-generation sensing and energy conversion devices.

Electronic Supplementary Material: Supplementary material (materials and reagents; apparatus; SEM image of CdS NRs (Fig. S1); TEM and HRTEM of CdS NRs (Figs. S2 and S3); TEM image of Au NPs (Fig. S4); XPS of CdS NRs, CdS_{QR}, Au–CdS_{QR} (Fig. S5); photoelectric properties (Fig. S6); EIS (Figs. S7 and S15); PL (Fig. S8); 3D surface potential distribution (Fig. S9); Tauc plots (Fig. S10); Mott–Schottky plots (Fig. S11); XPS valence band (Fig. S12); optimum reaction time (Fig. S13); feasibility verification (Fig. S14); selectivity (Fig. S16); anti-interference (Fig. S17); reproducibility (Fig. S18); the charge-transfer resistance (Tables S1 and S2); comparison of different CRP sensors (Table S3); detection of CRP in human serum samples (Table S4)) is available in the online version of this article at <https://doi.org/10.26599/NR.2026.94908857>.

Data availability

All data needed to support the conclusions in the paper are presented in the manuscript and the Electronic Supplementary Material. Additional data related to this paper may be requested from the corresponding author upon request.

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Declaration of competing interest

All the contributing authors report no conflict of interests in this work.

Author contribution statement

C. Z.: Data curation, methodology, formal analysis. M. W. L.: Conceptualization, writing – original draft, funding acquisition, project administration. X. W.: Data curation. W. H. Y.: Data curation. C. Z. Z. and A. G. S.: Writing – review & editing.

Use of AI statement

The authors used DeepSeek solely to polish the abstract's language; no AI was applied to other sections. All suggested edits were reviewed and finalized by the authors, who bear full responsibility for the entire manuscript.

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