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Efficiency Improvement of Photoelectrochemical Solar Cell Applications by Using Ternary Hybrid $MoS_2/g-C_3N_4/Cu_2O$

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Abstract. *In this paper, molybdenum disulfide (MoS_2) was hybridized with graphene carbon nitrite ($g-C_3N_4$) and Cu_2O in order to enhance the photoelectrochemical (PEC) activity and increase the light absorption range of Cu_2O thin film. The melamine powder was poured in an empty container and then heated in a furnace to attain the $g-C_3N_4$ powder. The ternary hetero-epitaxial growth was achieved by growing of $MoS_2/g-C_3N_4$ on the Cu_2O hybrid by a partial thermal oxidation process. The characteristics of $MoS_2/g-C_3N_4/Cu_2O$ hybrid film were investigated through XRD, FT-IR and photoelectronchemistry-related measurements. The PEC behavior of the ternary hybrid electrode was investigated using current-voltage test under illumination. The efficiency calculated from current-voltage test under illumination shows that the presence of graphene carbon nitrite and molybdenum disulphide within the film networks, despite its low content, could stimulate substantial improvement in maximum photoconversion efficiency from 0.036% to 0.33%. This improvement is attributable to the enhancement of the electron-transferring proficiency upon the insertion of $g-C_3N_4$ and MoS_2, as confirmed by X-Ray Diffraction Analysis (XRD). The PEC test results signify that the photoelectrochemical activity of the $MoS_2/g-C_3N_4/Cu_2O$ ternary hybrid is much higher than that of Cu_2O subtrate. The mechanisms accountable for the enhanced PEC behavior of the $MoS_2/g-C_3N_4/Cu_2O$ ternary hybrid are discussed in detail.*

Keywords: *Cuprous oxide, J-V characteristic, Hetero-structure, Photoelectrochemical, Thermal oxidation*

Introduction

The Industrial rebellion has brought about a fabulous increase in the world's population, and the ultimatum for energy is increasing [1]. It is anticipated that about 9 billion people will live on the planet by 2050, and about 30 TW of energy will be required to sustain this population. Nevertheless, more than 70% of our energy needs are currently met by finite fossil fuels, which will soon be exhausted [2-5]. Therefore, using a green backup energy source has become a big challenge for people. As a nearly limitless source of green energy, solar energy has received substantial attention in recent years. To this end, photoelectrochemical (PEC) cells are considered to be efficient devices for converting solar energy into hydrogen chemical energy by water splitting [6-7]. Photoelectrodes, usually made of semiconductors, play the most important roles in light absorption, electron-hole pairing and charge transfer in PEC cells [8-9]. However,

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due to their large bandgaps, most semiconductor materials can only absorb a small fraction of sunlight in the UV range, which severely limits their potential applications [10].

In order to improve the PEC efficiency of photoelectrodes, doping with compounds or elements and fabrication of semiconductors with heterostructures have been investigated in detail due to the different interactions between different semiconductor materials [11-12]. Among them, cuprous oxide (Cu_2O) has been considered as the state-of-the-art candidate of photocathode [13-14]. Cu_2O is a typical p-type semiconductor with a band gap of ~ 2 eV, with which it could achieve a theoretical photocurrent of -14.7 $mAcm^{-2}$ for water splitting and a solar to hydrogen conversion efficiency of 18.1 % on the AM 1.5 spectrum [14].

Moreover, it is scalable, earth-abundant, environmentally benevolent and compatible with inexpensive fabrication processes, which are important requirements to placate the terawatt-scale global energy demand [15]. The practical application of Cu_2O in the PEC process is still limited by two major drawbacks despite the aforementioned advantages: (1) sky-scraping recombination rate of photogenerated electron-hole carriers, partly ascribed to its mismatched electron diffusion length (usually 20–100 nm) with the light absorption depth (about 10 μm) [16]; (2) Deprived photostability because of self-photocorrosion in electrolyte solution [17].

Structure engineering has been reported to effectively address the above limitations of Cu_2O. Currently, owing to the simple preparation process, most of Cu_2O-based photocathode for PEC water splitting is built basing on Cu_2O film, usually showing a low photoelectric conversion efficiency [18-19]. In contrast, its Cu_2O nanowire/nanorod-based counterparts show significantly improved efficiency. This is mainly attributed to more efficient light harvesting, more efficient separation and transport of photogenerated charge carriers, larger surface area for fast interfacial charge transfer, and electrochemical reactions [20-21].

In addition to structure engineering, heterojunction engineering is widely considered as another effective strategy to improve PEC water splitting performance of Cu_2O through efficient separation of photogenerated charge carriers. [22-23]. The Cu_2O-bound semiconductor is not only a key element in forming the pn junction, but in some cases also acts as a protective layer that slows down the corrosion of the latter [24-25].

Owing to its layered structure, MoS_2 possesses highly exposed catalytic edge sites, while, g-C_3N_4 possess good visible-light absorption property and relatively high surface area [26]. However, the photocatalytic activity of pristine g-C_3N_4 has been found to be low owing to possible recombination of electron-hole pair. Therefore, for enhanced H_2 generation activity, g-C_3N_4 is normally coupled with other materials for effective charge separation [27-28].

Despite tremendous efforts, Cu_2O-based photocathode challenges still remain, and the combination of the above two strategies will lead to the development of a novel and efficient Cu_2O-based photocathode with potential applications in PEC water splitting. It indicates that the continuing need to further explore the photocathode could go further. This work deals with his PEC investigation of his Cu_2O films produced by thermal oxidation. Copper forms two different oxides, Cu_2O, with a direct bandgap of 2.1 eV [29-30], so it strongly absorbs only at wavelengths below 600 nm, whereas CuO with a bandgap of 1.21–1.51 eV [31-32] absorbs the entire visible range. In addition, other reasons for choosing Cu_2O as a material in this study are (a) its abundance in nature, (b) non-toxicity, (c) low cost of production, (d) stability and (e) fairly good

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electrical properties. The effects of ternary hybrid deposition of $MoS_2/g-C_3N_4/Cu_2O$ film's PEC behavior were investigated. In addition, the films were also characterized in terms of structure and phase discrimination using XRD, I-V characteristic curve and FTIR, respectively.

Materials and Methods

Synthesis of Cu_2O thin film

Commercial pure copper (99.98%) in foil form (0.1 mm thick) was cut into standard size wafers of 2 cm x 2 cm. The sample was pickled on the rim of the bottle to make it smooth, immersed in dilute nitric acid, rinsed thoroughly with distilled water several times, and then dried to remove impurities on the film surface. After cleaning, the copper film was thermally oxidized by furnace annealing in air. The oxidation temperature was controlled over a wide range from room temperature (RT) to 450 °C. The heating rate was about 10 °C./min, and once the preferred maximum temperature was reached, it was held for 30 minutes to allow copper oxide to form. After oxide formation, the furnace was allowed to cool for 2 hours. Slow cooling was maintained to minimize possible thermal stress and film cracking.

Preparation of g-C_3N_4

In a typical synthesis, the g- C_3N_4 photocatalyst was synthesized separately from melamine [33]. 3.0 g of melamine was taken in alumina crucibles with cover and calcined at 500 °C with a heating rate of 3 °C min^{-1}. It is further heated to 520 °C for 2 h at a heating rate of 2 °C min^{-1} and allowed to cool down to room temperature. A yellow product was collected and ground into fine powder. The sample was named as CN.

Synthesis of $\boldsymbol{MoS_2/g-C_3N_4/Cu_2O}$ hybrid film

The method used to grow MoS_2 was chemical vapor deposition (CVD) [34-35]. The substrate was placed under specific temperature and pressure conditions and one or more precursors were chemically reacted on the surface of the substrate to produce a high-quality large-area thin film. The application of CVD in the preparation of single-layer TMDs starts with MoS_2 growth. The heating rate was set to 10 °C per min, the growth temperature was 650 °C, and the temperature was held for 30 min. Once the temperature was dropped to 400 °C, the lid was opened. The MoS_2 structure was obtained when the temperature decreases to room temperature. The experimental process was shown in **Figure 1**. CVD can effectively produce monolayer and multilayer $MoS_2/g-C_3N_4$. It was able to grow high-quality single-crystal materials and produce thin films uniformly distributed over large areas, which were useful in later fabrication of optoelectronic components.

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Figure 1. Schematic of a growing $MoS_2/g-C_3N_4/Cu_2O$ film.

Photoelectrochemical tests

For this purpose, $MoS_2/g-C_3N_4/Cu_2O$ and Cu electrode were arranged and dipped into transparent plastic container. To prepare the electrolyte, the 1 g of NaCl powders were mixed with 25 ml of distilled water, stirrer gently until the electrolyte was dissolved completely. The PEC performance of the hybrid electrodes was evaluated using Current-Voltage measurements (**Figure 2**). The photoelectrochemical tests were performed via a two-electrode electrochemical system. A working electrode ($MoS_2/g-C_3N_4/Cu_2O$) and a copper plate were employed as the counter electrodes, respectively. The multimeter was employed to accomplished electrical route of the photocurrent density and photo voltage of electrodes under illumination (AM 1.5 G) within the potential window using 1 g of NaCl electrolyte as a mediator between the two electrodes. The approach described in this study provides a simple and novel method to synthesize thin film materials, ready for practical applications such as the photoelectrochemical solar cell and hydrogen production.

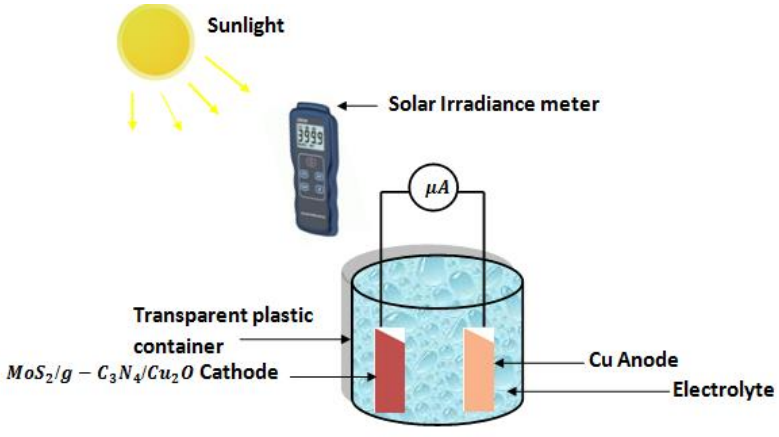

Figure 2. Illustration of the fabricated Cu – $MoS_2/g-C_3N_4/Cu_2O$ Photoelectrochemical solar cell

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Results and Discussion

Structural analysis

The crystal structure and the crystalline nature of the thermally oxidize $MoS_2/g-C_3N_4/Cu_2O$ films were investigated by XRD analysis. **Figure 3** shows the XRD patterns of $MoS_2/g-C_3N_4/Cu_2O$ thin films deposited at different thermally oxidized temperature. XRD patterns reveal that the deposited Cu_2O films are polycrystalline in nature and belong to cubic structure. In addition, all slightly increasing characteristic peaks from the ternary samples were been examine entirely and their peaks are at 2θ values of 29.01°, 36.52°, 42.11° and 61.11° corresponding to (110), (111), (200) and (220) diffraction plane of Cu_2O (JCPDS card no. 05-0667), respectively, were detected [36-38]. Thermally oxidized MoS_2 the observed peaks at 33.6° correspond to (101) 37.158° display peak corresponding to (212) plane of $g-C_3N_4$, [39-42]. 38.9°, 43.8° and 52.2° corresponding to CuO (111), Cu (111) and Cu (200) [38]. MoS_2 also displays peaks that correspond to (101) at 33.4°, (104) at 46.1° and (008) at 61.7° [43-44]. **Table 1** and **Figure 4** displayed the analysis result of the ternary sample.

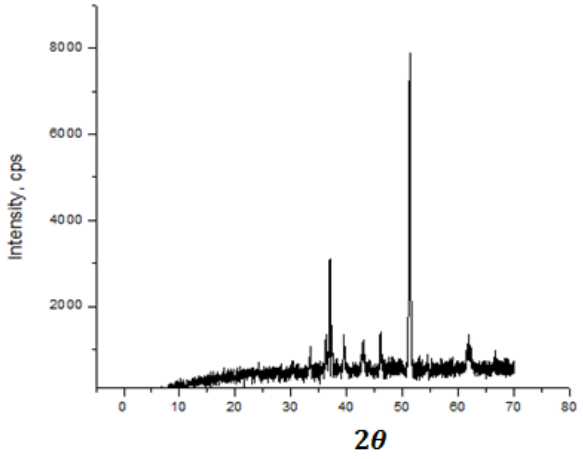

Figure 3. XRD pattern of MoS_2/g-C_3N_4/Cu_2O

Table 1. Analysis Results of the sample

No.	Phase name	Formula	Figure of merit
1	Cuprite	Cu_2O	0.950
2	Molybdenite-2H	MoS_2	2.970
3	Copper	Cu	2.448
4	Iron	Fe	1.003

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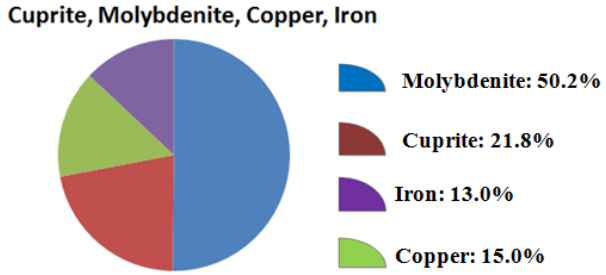

Figure 4. Analysis report of the synthesized sample

FT-IR spectra analysis

FT-IR analysis **(Figure 5**) was performed to analyze the chemical and structural properties of bare g-C_3N_4, MoS_2, and Cu_2O thin films. Pure g-C_3N_4 exhibits a characteristic IR peak at 1632 cm^{-1}, whereas peaks at 1245, 1320 and 1417 cm^{-1} are assigned to the C-N heterocyclic stretch of g-C_3N_4 [45-46]. Broad shoulder bands in the regions of 3150, 3320 and 3350 cm^{-1} correspond to the stretching modes of the terminal NH groups at the aromatic ring defects [44-45]. The presence of oxygen-related bonds was due to the presence of sharp bonds about 3620 cm^{-1} [47-48]. The peak at 3405 cm^{-1} is attributed to O-H groups [47-48], and 839 cm^{-1} and 893.39 cm^{-1} are broad absorption bands attributed to MoS_2 [49-50]. The peak at 3506 cm^{-1} is due to the oxygen related compound [47-48]. A peak of about 1730 cm^{-1} indicates the presence of C = O bonds in the sample. The 2325-2425 cm^{-1} band represents the P-H stretch. 2998-2959 cm^{-1} is assigned to symmetric C-H stretch vibration. From **Figure 5** it is observed that, the characteristic peaks for Cu_2O (630 cm^{-1}) shift (towards lower wavenumber) after the incorporation of g-C_3N_4 and MoS_2, which indicated that there was an interaction between g-C_3N_4, Cu_2O and MoS_2. From the spectra, all the characteristic peaks of both g-C_3N_4, Cu_2O and MoS_2 appeared in the MoS_2/g-C_3N_4/Cu_2O, which is in accordance with the Fourier infrared spectra.

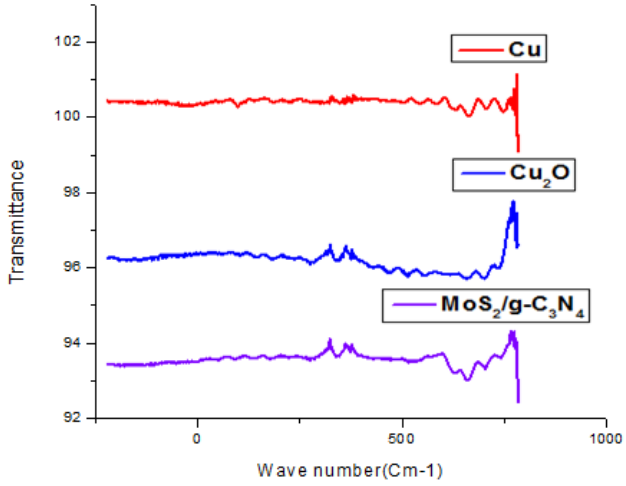

Figure 5. FTIR spectra of $MoS_2/g-C_3N_4/Cu_2O$

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I-V Curve analysis

The efficiency, maximum power, photo voltage and photocurrent was obtained under illumination as outline in **Table 2** and the Cu- $MoS_2/g-C_3N_4/Cu_2O$ PEC solar cell showed characteristic curves of a number of external parameters followed by transition power efficiency deduce from **Figure 6** and **Figure 7**. When analyzing the prototype, the calculated external parameters of the prepared samples Cu-Cu_2O and $MoS_2/g-C_3N_4/Cu_2O$ are stated as shown in **Table 2**. Two different readings are recorded using a multimeter and numerous solar irradiances in order to study the solar cell parameters, two dissimilar graphs are studied for two different samples for testing photo response and photo voltage of the electrode under illumination. In **Table 2** it can be seen that for the synthesized $MoS_2/g-C_3N_4/Cu_2O$, the deposited of 2D materials increases the photo response at the same time increasing the efficiency of the sample. It also works as an absorber layer to generate charge carriers (electrons and holes) under solar light irradiation. A Comparison of some Findings of I_{sc}, V_{oc} and η with other Works in Literature displayed in **Table 3** below.

Table 2. The photocurrent, maximum power, efficiency and photo voltage of dissimilar reading of Cu-Cu_2O and Cu- $MoS_2/g-C_3N_4/Cu_2O$ photoelectrochemical solar cell

S/N	Sample	$I_{sc}(mA)$	$V_{oc}(mV)$	$\eta(\%)$
1	Cu_2O	0.14	11.0	0.036
2	$MoS_2/g-C_3N_4$	1.20	60	0.33

The first row in **Table 2** is for Cu-Cu_2O synthesized using thermal oxidation method while the subsequent row is for - $MoS_2/g-C_3N_4/Cu_2O$ layer deposited by partially thermal oxidation method.

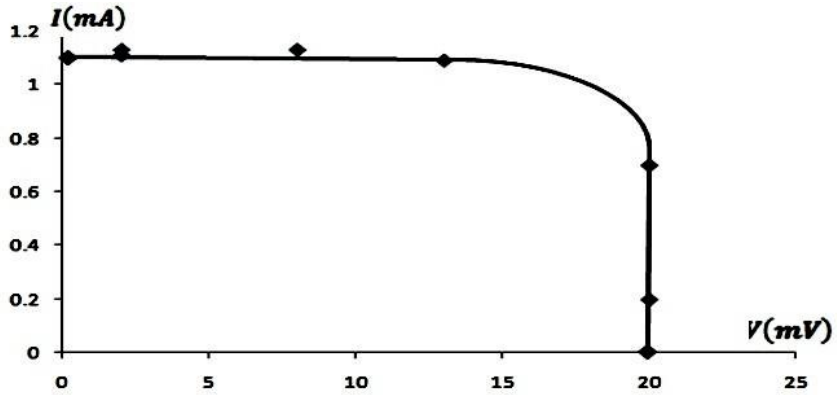

Figure 6. The graph of Cu-Cu_2O photoelectrochemical solar cell before surface modification

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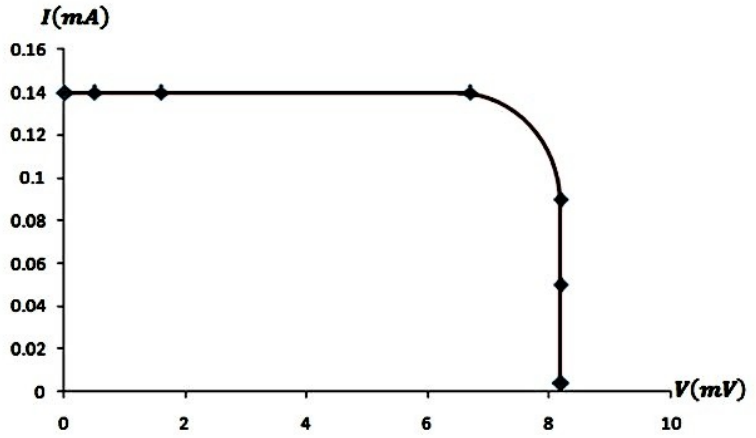

Figure 7. The graph of Cu- $MoS_2/g-C_3N_4/Cu_2O$ photoelectrochemical solar cell after surface modification

One can see from **Figure 6** and **Figure 7** that there is change in all the solar cells external parameters can be enhancement which is attained for lengthy wavelength light, when majority of electron-hole pairs are generated outside of the space charge region (SCR) and high lifetime values are required for participation of carriers in PEC reactions. On the other hand, the similar enhancement of power conversion efficiency due to deposition of $MoS_2/g-C_3N_4$ adding up to the thermal oxidation is observed also for a short wavelength light, which is absorbed mainly in the thin near-surface part of film as shown in **Table 2** and **Table 3**.

Table 3. Comparison of Findings with other Works in Literature

Authors (Year)	Structures	Method	Efficiency	Findings	Ref.
J. Herion et al. (1979)	Cu_2O		1%	V_{oc}	[51]
	Cu/Cu_2O	Partial thermal oxidation	0.4%		
R. P. Wijesundera (2010)	$Ti/CuO/Cu_2O/Au$	Electro-deposition	0.02%	*FF*, I_{sc},V_{oc}	[52]
Katayama et al. (2004)	$Cu_2O/ZnO/ITO$	Electro-deposition	0.117%	*FF*, I_{sc},V_{oc}	[53]
Septina et al. (2011)	Cu_2O/AZO	Electro-deposition	0.60%	*FF*, I_{sc},V_{oc}	[54]
Seyed, A.J (2013)	Cu_2O	Electro-deposition	0.082%	*FF*, I_{sc},V_{oc}	[55]
Y.-K. Hsu et al. (2015)	Cu_2O	Electro-deposition	0.42	*FF*, I_{sc},V_{oc}	[56]
Abdu Y *(2017)*	Cu_2O	Thermal oxidation	0.08	*FF*, I_{sc},V_{oc}	[57]
Vijayaraghavan et al. (2018)	CdTe	SPD technique	0.062	*FF*, I_{sc},V_{oc}	[58]
Roza et al. (2014)	ZnO	Hydrothermal	0.050	*FF*, I_{sc},V_{oc}	[59]
Abdurrahman (2019)	Cu_2O	Thermal oxidation	0.046	*FF*, I_{sc},V_{oc}	[60]

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Authors (Year)	Structures	Method	Efficiency	Findings	Ref.
Sutripto et al. (2019)	CdO	chemical method	0.21	*FF, I_{sc},V_{oc}*	[61]
Tadatsugu Minami et al. (2016)	Zn_2GeO_4	Thin film deposition	0.12	*FF, I_{sc},V_{oc}*	[62]
Tadatsugu Minami et al. (2016)	Zn_2SiO_4	Thin film deposition	0.03	*FF, I_{sc},V_{oc}*	[62]
Tadatsugu Minami et al. (2016)	$ZnSnO_3$	Thin film deposition	0.01	*FF, I_{sc},V_{oc}*	[62]
This work		Thermal oxidation	0.33	*FF, I_{sc},V_{oc}*	
Abdurrahman (2022)	Cu_2O	Thermal oxidation	4.80	*FF, I_{sc},V_{oc}*	[4]

Conclusions

A ternary hybrid composed ofMoS_2, $g-C_3N_4$ and Cu_2O thin film was synthesized for photoelectrochemical solar cell applications. The $MoS_2/g-C_3N_4$ binary hybrid was prepared by partial thermal oxidation processing of MoS_2 and $g-C_3N_4$ in a sealed high temperature furnace. The epitaxial growth of MoS_2 and that of $g-C_3N_4$ on the surfaces of Cu_2O thin film were taken place during the partial thermal oxidation process. Full structure and PEC analysis indicate that the enhanced PEC activity could be attributed to the synergy between the two 2D materials. In addition, 2D materials exhibit a passivation effect that not only improves photo-excitation voltage and current by reducing the recombination rate of charge carriers, but also increases surface photoelectrochemical to enhance the photocurrent by rushing the separation of the surface charge and utilization. The corresponding stronger electric filed strength in the space charge layer significantly increases the separation efficiency of photogenerated electron-hole pairs and eventually improves their PEC performances. The synthesized $MoS_2/g-C_3N_4/Cu_2O$ showed a higher efficiency of 0.33% than that of the Cu_2O 0.036% sample.

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