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Investigation of One-Dimensional PAN/SrTiO₃@Al Composite Nanofibers for Hydrogen Evolution

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Abstract

The development of promising photocatalysts for hydrogen production is one of the most important areas of research. Enhanced efficiency can be achieved by modifying the composition and creating composite structures, thereby improving the separation of photogenerated charge carriers. In this work, SrTiO₃ (STO) and Al-doped SrTiO₃ (SrTiO₃@Al; hereafter denoted as ASTO) photocatalysts were synthesized, followed by the incorporation of Rh-, Cr-, and Co-containing cocatalysts to obtain ASTO@Rh/Cr/Co. Polyacrylonitrile (PAN)-based composite nanofibers containing the corresponding photocatalysts were prepared by electrospinning followed by carbonization. The synthesized materials were characterized by scanning electron microscopy (SEM) and Fourier-transform infrared (FTIR) spectroscopy, while their photoelectrochemical properties were evaluated using photocurrent measurements. The photocatalytic properties of the composites were investigated in hydrogen evolution reactions. The hydrogen evolution rate of the PAN/ASTO@Rh/Cr/Co composite was found to be 319.5 μmol·g⁻¹·h⁻¹, which is 1.96 times higher than that of PAN/STO and 6.64 times higher than that of the PAN/ASTO composite. These results demonstrate that the incorporation of Rh/Cr/Co cocatalysts into the PAN/ASTO composite substantially enhances photocatalytic hydrogen evolution under the investigated experimental conditions.

1. Introduction

Currently, global climate and environmental changes, together with increasing energy consumption and the depletion of energy resources, are driving the search for alternative energy sources and the development of sustainable, environmentally friendly technologies for energy production [1, 2]. In this context, hydrogen energy has attracted particular attention, as hydrogen (H₂) is considered one of the most promising environmentally friendly energy carriers of the future [3–6]. Depending

on the production method, hydrogen is commonly classified by color. The most widely produced type is “gray” hydrogen, which is obtained through the steam reforming of natural gas, whereas the production of “blue” hydrogen additionally involves the capture and storage of the resulting CO₂. However, both production routes remain associated with carbon dioxide emissions [7–11]. In contrast, “green” hydrogen refers to hydrogen produced through water electrolysis powered by renewable energy sources, particularly solar energy, with minimal carbon emissions throughout the production process [12–16]. In this regard, photocatalytic water splitting driven by solar irradiation represents one of the most promising approaches to green hydrogen production [17–20]. Among various photocatalytic

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materials, SrTiO₃ (STO), an oxide with a perovskite structure, is of particular interest because of its high photocatalytic activity and excellent physicochemical stability [21–25].

Despite these advantages, the application of STO as a photocatalyst is limited by several factors, including its wide bandgap (3.2 eV), which restricts light absorption to the ultraviolet region of the spectrum, and the high recombination rate of photogenerated electron-hole pairs. To overcome these limitations, various modification strategies have been employed, including doping with metal ions [26–28] and non-metals [29], deposition of cocatalysts [30], and the fabrication of heterostructures with narrow-bandgap semiconductors to form Z-scheme systems [31–34]. In particular, doping with Al³⁺ ions can minimize undesirable recombination centers and optimize the defect structure of STO [35–39]. Furthermore, incorporation of metal cocatalysts promotes efficient charge separation, thereby significantly enhancing the photocatalytic performance of the system [40, 41]. Another promising approach involves the fabrication of heterojunction-based composite materials by electrospinning, a method that has attracted considerable attention due to its simplicity and accessibility [42, 43]. This technique enables the production of continuous one-dimensional nanofibers with a high specific surface area, porous structure, and numerous active sites [44–48]. In particular, a previous study [49] demonstrated that the use of PAN fibers as a matrix for the g-C₃N₄/Bi₂Sn₂O₇ composite enhances photocatalytic hydrogen evolution by improving the separation and transport of charge carriers.

Previous studies have reported STO/PAN-based composite nanofibers [50, 51]; however, their application and comparative performance in photocatalytic H₂ evolution remain insufficiently investigated. Therefore, in this study, PAN/STO, PAN/ASTO, and PAN/ASTO@Rh/Cr/Co composite nanofibers were prepared by electrospinning, followed by carbonization and systematically evaluated for photocatalytic H₂ evolution under identical experimental conditions.

2. Experimental part

2.1 Materials

Strontium nitrate (Sr(NO₃)₂, ≥98%, Sigma-Aldrich), titanium (IV) oxide, anatase (TiO₂, 99.9%, MERYER), oxalic acid ((COOH)₂, 99%, MERYER), ultrapure water (Elga Purelab Quest UV), aluminium

oxide (Al₂O₃, Sigma-Aldrich), rhodium (III) chloride hydrate (RhCl₃·6H₂O, Rh content 38–40%), potassium chromate (K₂CrO₄), cobalt (II) nitrate hexahydrate (Co(NO₃)₂·6H₂O), N,N-dimethylformamide (DMF, ≥99.8%, Sigma-Aldrich), polyacrylonitrile (PAN) average molecular weight approximately 1,300,000 g/mol, lactic acid (C₃H₆O₃, Sigma-Aldrich).

2.2 Synthesis of STO, ASTO, and ASTO@Rh/Cr/Co

The STO, ASTO, and ASTO@Rh/Cr/Co samples were synthesized according to previously reported methods [50, 51]. Briefly, the STO samples were prepared by chemical precipitation followed by calcination. ASTO samples were synthesized using a flux method with the addition of 2 mol.% Al₂O₃ relative to STO.

The cocatalysts were deposited onto the ASTO surface by photodeposition. For the preparation of ASTO@Rh/Cr/Co, 50 μL of RhCl₃·6H₂O solution (2 mg/mL) was first added to the ASTO suspension, followed by photodeposition for 10 min. Subsequently, 25 μL of K₂CrO₄ solution (2 mg/mL) was added, and photodeposition was continued for 5 min. Finally, 25 μL of Co(NO₃)₂ solution (2 mg/mL) was added, followed by an additional 5 min of photodeposition. The total photodeposition time was 20 min. The resulting samples were washed and dried at 60 °C.

2.3 Synthesis of PAN/STO, PAN/ASTO, and PAN/ASTO@Rh/Cr/Co composite nanofibers

The composite nanofibers were prepared by electrospinning according to the method described in our previous studies [35, 51]. Briefly, 0.7 g of PAN was dissolved in 7 mL of DMF under magnetic stirring at 450 rpm and 50 °C for 30 min. The corresponding photocatalyst was then added to the resulting solution at a PAN to photocatalyst mass ratio of 1:1, and the mixture was stirred for an additional 30 min to ensure uniform dispersion. The resulting electrospinning solution was transferred into a 5 mL syringe. Electrospinning was performed at an applied voltage of 18 kV, a solution flow rate of 1 mL·h⁻¹, and needle-to-collector distance of 15 cm. The resulting nanofibers were dried at 60 °C for 12 h and subsequently subjected to a two-stage heat-treatment process. First, the nanofibers were thermally stabilized at 270 °C for 60 min, followed by carbonization at 800 °C for 120 min under an inert argon atmosphere.

2.4 Characterization

The surface morphology and elemental composition of the composite nanofibers before and after carbonization were investigated by scanning electron microscopy using a Zeiss Crossbeam 540 microscope (ZEISS, Germany) and a JEOL JSM-6510LV microscope (JEOL, Japan). The SEM measurements were performed at accelerating voltages ranging from 5 to 20 kV. The FTIR spectra of the samples were recorded using a WQF-530A spectrometer.

2.5 Photoelectrochemical measurements

The photoelectrochemical measurements were performed in a three-electrode cell using a CorrTest CS310M potentiostat (CorrTest Instruments, China). The working electrode was prepared by drop-coating a suspension of 5 mg of the photocatalyst in a mixture of isopropanol and Nafion onto pre-cleaned FTO glass with an active area of 1 cm², followed by drying at 60 °C. A Pt electrode and an Ag/AgCl electrode were used as the counter and reference electrodes, respectively, while 0.5 M Na₂SO₄ was used as the electrolyte. Irradiation was provided by a 300 W Xe lamp equipped with a solar simulator. Transient photocurrent measurements were performed at an applied potential of 1.0 V vs. Ag/AgCl using alternating light–dark cycles of 20 s. Electrochemical impedance spectroscopy (EIS) measurements were performed at 0.5 V vs. Ag/AgCl over a frequency range from 100 kHz to 0.01 Hz with an AC amplitude of 10 mV under both dark and illuminated conditions. Mott–Schottky measurements were performed in the dark over a potential range from –1.2 to +1.2 V vs. Ag/AgCl at a frequency of 1 kHz with an AC amplitude of 10 mV. The resulting 1/C² versus potential plots were used to determine the flat-band potentials and conductivity type of the samples.

2.6 Photocatalytic Hydrogen Evolution

The photocatalytic hydrogen evolution activity of the composite nanofibers in the presence of a sacrificial agent was evaluated using a Trace Gas Reaction Evaluation System (μGAS1000, Beijing Perfectlight Technology Co., Ltd.) equipped with a 300 W xenon lamp as the light source. The amount of evolved hydrogen was quantified using a TETREK GC2030 gas chromatograph connected to the system. The experiments were conducted in a 50 mL glass reactor. The photocatalyst (40 mg) was dispersed in 45 mL of an aqueous solution by ultrasonication for 10 min, after which lactic acid (10 vol%), serving as a sacrificial

electron donor, was added. The temperature of the reaction mixture was maintained at 5 °C using a water-cooling system. Prior to irradiation, the system was evacuated and purged with argon to remove residual air and establish an inert atmosphere. The composition of the evolved gases was analyzed every 60 min by gas chromatography using argon as the carrier gas and a thermal conductivity detector (TCD) and a flame ionization detector (FID).

3. Results and Discussion

3.1. Characterization

3.1.1 SEM

The surface morphology of the synthesized composite materials was analyzed using SEM. Figure 1 shows SEM images of PAN/STO nanofibers (a, b) and PAN/ASTO@Rh/Cr/Co nanofibers (c, d) before and after heat treatment. Prior to heat treatment, the samples are characterized by a randomly oriented one-dimensional fibrous structure with a relatively smooth surface. For PAN/STO, the mean fiber diameter is approximately 214 ± 52 nm (Fig. S2a). Light-colored areas and isolated agglomerates of particles are observed on the fiber surface, which may be associated with the presence of an inorganic component in the composite structure [35, 50].

After heat treatment, the morphology of the PAN/STO fibers changes significantly: the fibers become partially fused and form a more interconnected porous network, while the mean fiber diameter increases to approximately 553 ± 189 nm (Fig. S2b). The surface becomes rougher and local thickening of the fibrous structure is observed, indicating thermally induced restructuring and partial fusion of the fibers during carbonization of the polymer matrix. PAN/ASTO@Rh/Cr/Co composite fibers also retain a one-dimensional morphology with a mean fiber diameter of approximately 198 ± 59 nm (Fig. S2c) prior to heat treatment. After heat treatment, the fibrous framework remains distinguishable, although partial fusion, local agglomeration, and increased surface roughness are observed. The mean fiber diameter increases only slightly to approximately 229 ± 67 nm (Fig. S2d). The statistical fiber-diameter distribution was determined by measuring 70 individual fibers for each sample from the corresponding SEM images, and histograms were constructed to compare the diameter distributions (Fig. S2). Thus, in contrast to PAN/STO, thermal treatment causes only a minor change in the mean diameter of PAN/ASTO@Rh/Cr/Co fibers, while the main morphological changes are

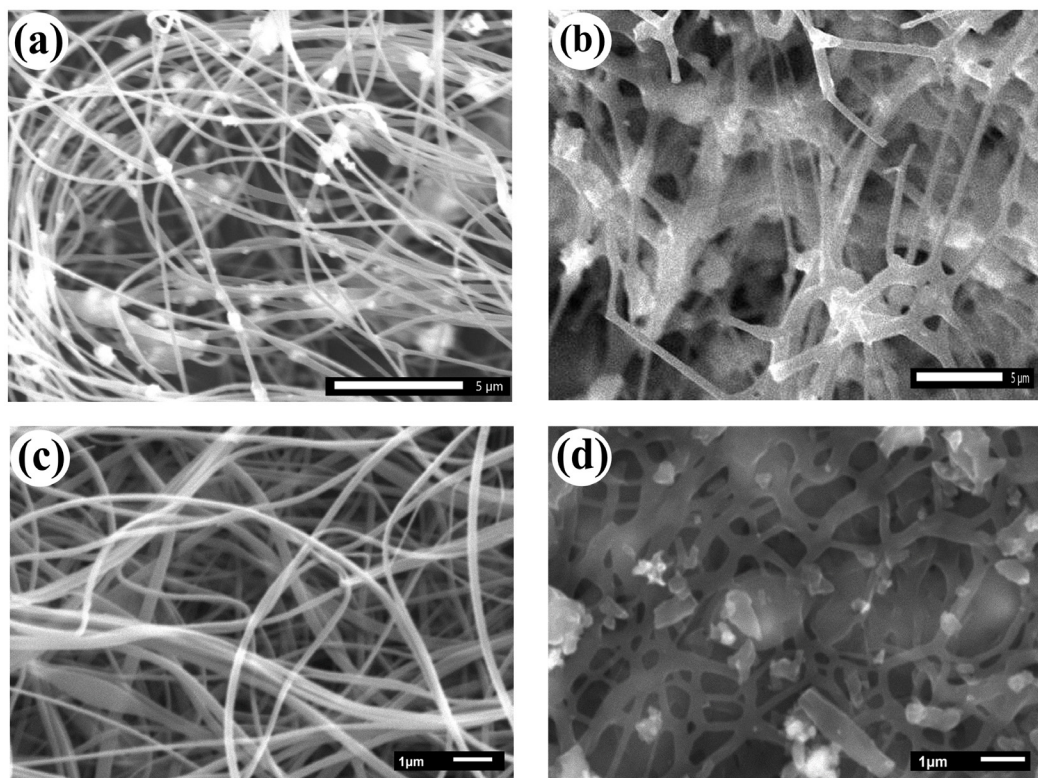


Fig. 1. SEM images of PAN/STO (a, b) and PAN/ASTO@Rh/Cr/Co (c, d) nanofibers before and after thermal treatment.

associated with surface restructuring and partial interconnection of the fibers. The results of energy dispersive X-ray analysis (EDX) supported the elemental composition of the obtained samples. For the PAN/STO sample (Fig. 2a) C, O, Ti and Sr elements were detected, with contents of 90.44%, 0.71%, 4.57% and 4.28%, respectively (Table S1). The EDX analysis revealed no additional impurity elements within the detection limit of the method.

In the PAN/ASTO@Rh/Cr/Co composite, C, N, O, Sr, and Ti signals were observed, while weak trace-level signals associated with Al, Cr, and Rh

were also recorded. These trace-level signals were interpreted cautiously because of the limited reliability of EDX for quantitative analysis at very low concentrations, whereas Co was not considered reliably detected (Table S2).

3.1.2 FTIR

As shown in Fig. 3, the FTIR spectra of PAN, PAN/STO, PAN/ASTO, and PAN/ASTO@Rh/Cr/Co exhibit differences in the positions and intensities of the absorption bands, which are most pronounced in the low-wavenumber region. The broad band in

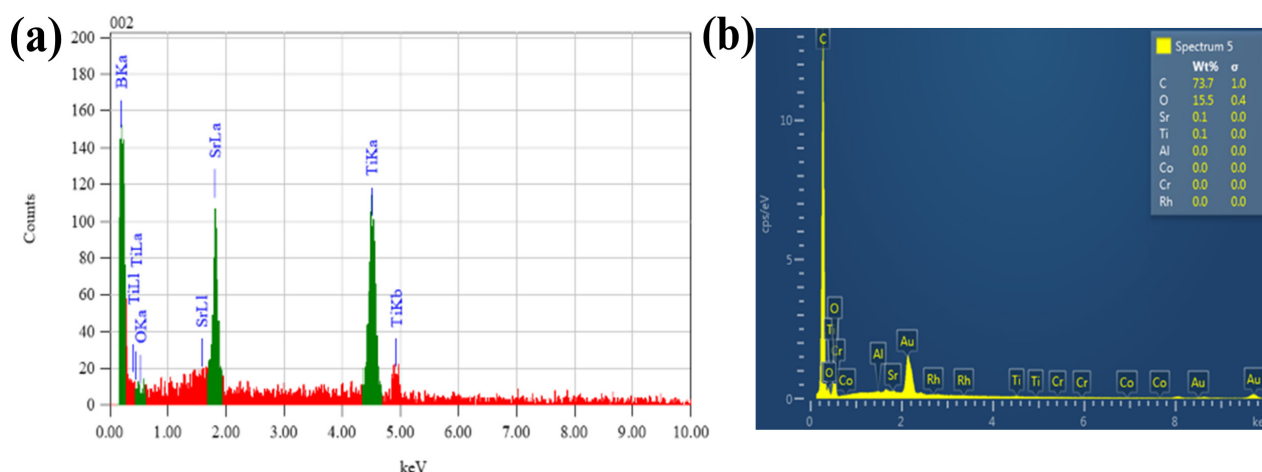


Fig. 2. EDX mapping of PAN/STO (a) and PAN/ASTO@Rh/Cr/Co (b).

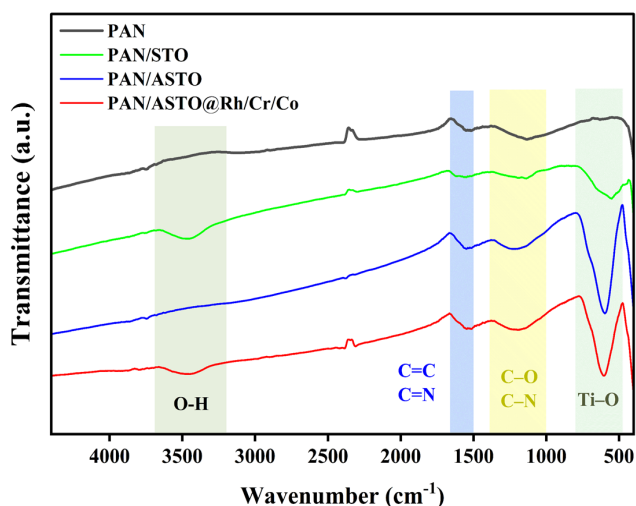


Fig. 3. FTIR spectra of thermally treated PAN/STO, PAN/ASTO, and PAN/ASTO@Rh/Cr/Co composites.

the range of 3200–3600 cm^{-1} is attributed to the stretching vibrations of O–H groups associated with surface hydroxyl species and adsorbed water [53]. Weak bands in the 1500–1670 cm^{-1} region may be associated with C=C and C=N vibrations of the carbon structure, as well as with the bending vibrations of adsorbed water [54]. The bands in the 1000–1390 cm^{-1} region may be related to C–O and C–N vibrations of residual surface functional groups in the carbon matrix [55].

The most significant spectral differences among the samples are observed in the 470–800 cm^{-1} region. In the spectra of PAN/ASTO and PAN/ASTO@Rh/Cr/Co, an intense band centered at approximately 600 cm^{-1} is observed, which is predominantly attributed to Ti–O vibrations in TiO_6 octahedra of the SrTiO_3 perovskite structure [53]. In the PAN/STO spectrum, the corresponding band is considerably weaker. The introduction of Rh/Cr₂O₃ and CoOOH cocatalysts is accompanied by changes in the intensity and a slight shift of the band near 600 cm^{-1} , which may be asso-

ciated with additional contributions from Cr–O and Co–O vibrations overlapping with Ti–O vibrations, as well as with changes in the local environment of the metal–oxygen bonds [35, 56].

Thus, the most pronounced changes in the FTIR spectra occur in the metal–oxygen vibration region and are mainly manifested as variations in band intensity and slight shifts in absorption-band positions. At the same time, these spectral features alone do not establish the preservation of the crystalline SrTiO_3 phase after carbonization. Meanwhile, the retention of the main spectral features associated with the carbon matrix suggests that no substantial changes occurred in its surface functional groups.

3.1.3 Photocurrent

Transient photoelectric responses (Fig. 4a) show that the PAN/ASTO@Rh/Cr/Co sample is characterized by a higher photocurrent density compared to PAN/ASTO, indicating efficient separation and transport of photogenerated charge carriers. The enhanced photoelectrochemical response of PAN/ASTO@Rh/Cr/Co may be associated with the combined effect of Rh/Cr/Co cocatalyst modification on interfacial charge transfer and charge-carrier separation. According to previous studies, Rh-, Cr-, and Co-containing cocatalysts can participate in charge-transfer and surface redox processes [57–59]. However, since samples containing Rh, Cr, and Co individually were not investigated in the present study, the specific contribution of each cocatalyst cannot be distinguished from the current experimental data.

The electrochemical impedance spectroscopy results (Fig. 4b) further support this conclusion. The PAN/ASTO@Rh/Cr/Co sample exhibits the lowest charge transfer resistance at the semiconductor/electrolyte interface, whereas the PAN/ASTO sample exhibits the highest interfacial resistance.

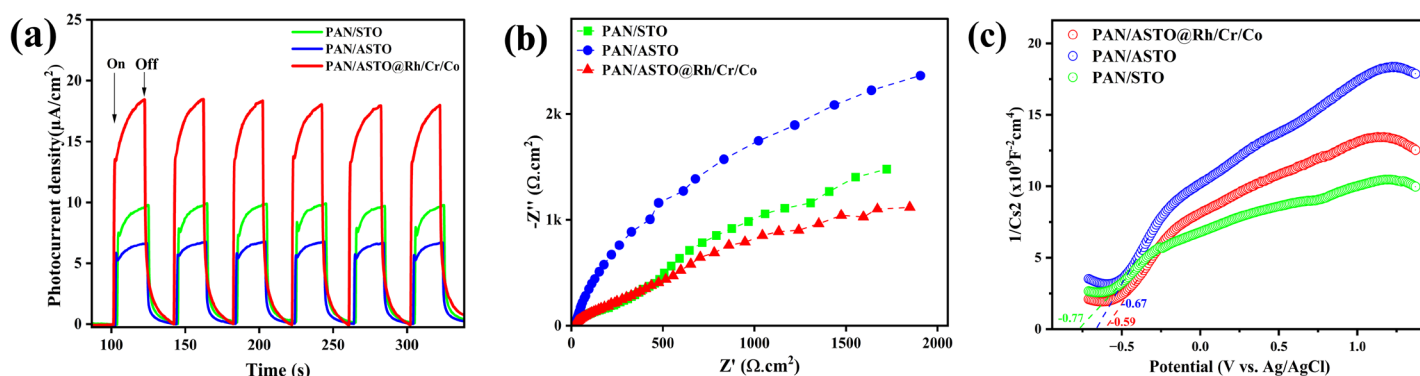


Fig. 4. Photoelectrochemical characteristics: transient photocurrent responses (a), photoelectrochemical impedance spectra (b) and Mott-Schottky plots (c).

This decrease indicates enhanced interfacial electron transfer and a reduced probability of photo-generated charge-carrier recombination. According to the Mott–Schottky analysis (Fig. 4c), the positive slope of the linear regions of the $(1/C^2)$ dependence on potential curves for all samples confirms that they are n-type semiconductors. Extrapolation of the linear regions onto the potential axis allowed us to determine the flat-band potentials, which are -0.77 V, -0.67 V, and -0.59 V (relative to Ag/AgCl) for PAN/STO, PAN/ASTO, and PAN/ASTO@Rh/Cr/Co, respectively. The potential shift toward the positive side following the combined Rh/Cr/Co modification indicates a change in the position of the energy bands near the semiconductor surface.

This might enhance band bending and contribute to more efficient separation of photogenerated charge carriers.

3.2. Photocatalytic Efficiency

The photocatalytic efficiency of the obtained composite materials was evaluated by photocatalytic hydrogen evolution tests under simulated solar irradiation. The PAN/STO sample exhibited a hydrogen evolution rate of $169.3 \pm 3.51 \mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$. In contrast, PAN/ASTO resulted in a decrease in the hydrogen evolution rate to $48.1 \pm 0.192 \mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$. The incorporation of Rh/Cr/Co cocatalysts enhanced the photocatalytic performance, resulting in the highest H₂ evolution rate of $319.5 \pm 3.88 \mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$ for PAN/ASTO@Rh/Cr/Co (Fig. 5a, b). The photocatalytic H₂-evolution experiments were independently repeated twice ($n = 2$) under identical experimental conditions to assess repeatability of the obtained results. The reported H₂-evolution rates were calculated from the experimental measurements, and

the error bars shown in Fig. 5b represents the standard deviation (SD) of the two independent measurements. This decrease is consistent with the photoelectrochemical results, which showed a lower photocurrent density and higher charge-transfer resistance for PAN/ASTO, indicating less efficient separation and transport of photogenerated charge carriers. The incorporation of Rh/Cr/Co cocatalysts was associated with enhanced photocatalytic performance.

The amount of evolved H₂ was determined using the calibration curve:

$$y = 41090x + 3383.7, R^2 = 0.9885,$$

where y is the GC peak area and x is the injected volume of H₂ (mL).

The measured H₂ volume was subsequently converted to the amount of H₂ (μmol) using the ideal gas equation. Based on the obtained data, the H₂ evolution rate was calculated using the following equation:

$$r_{H_2} = \frac{n_{H_2}}{m \times t}$$

where n_{H_2} is the amount of H₂ produced (μmol), m is the photocatalyst mass (g), and t is the irradiation time (h). The obtained values were expressed in $\mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$.

To evaluate the results more objectively, a comparative analysis of PAN/ASTO@Rh/Cr/Co was conducted against previously published one-dimensional photocatalysts for the H₂ evolution reaction (Table 1). It should be noted that the values are presented in Table 1 for photocatalysts and direct quantitative comparison is limited by differences in sacrificial agents, catalyst loading and experimental reactor configurations.

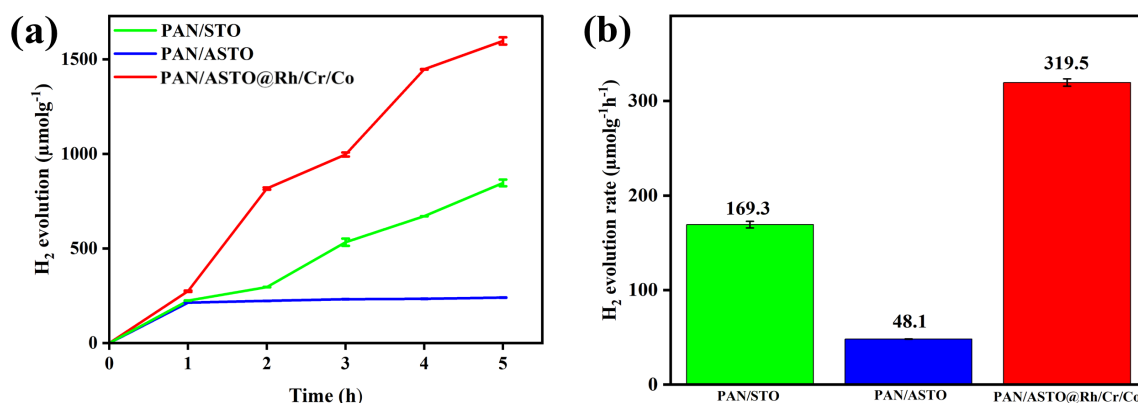


Fig. 5. Photocatalytic H₂ evolution on PAN/STO, PAN/ASTO, and PAN/ASTO@Rh/Cr/Co samples: cumulative H₂ evolution as a function of irradiation time (a); H₂ evolution rates (b).

Table 1. Comparison of the photocatalytic activity of PAN/ASTO@Rh/Cr/Co with previously reported one-dimensional photocatalysts in the H₂ evolution reaction

Photocatalyst	Sacrificial agent	Light source	H ₂ evolution rate/ ($\mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$)	Catalyst amount	Ref.
SrTiO ₃	–	300 W Xe-lamp	72	50 mg	[60]
PAN	None	300 W Xe-lamp	240	none	[47]
Pt/SrTiO ₃	Methanol aqueous solution	300 W Xe-lamp	35	50 mg	[61]
Mo-doped ZnIn ₂ S ₄	10 mL lactic acid	300 W Xe-lam	138.8	20 mg	[62]
Cu@g-PAN/STO	TEOA, 10%	300 W Xe-lamp	265.25	50 mg	[48]
PAN/STO	45 mL aqueous solution + 10 vol.% lactic acid	300 W Xe-lamp	169.3	40 mg	This work
PAN/ASTO	45 mL aqueous solution + 10 vol.% lactic acid	300 W Xe-lamp	48.1	40 mg	This work
PAN/ASTO@Rh/Cr/Co	45 mL aqueous solution + 10 vol.% lactic acid	300 W Xe-lamp	319.5	40 mg	This work

For SrTiO₃, the hydrogen evolution rate was 72 $\mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$ [60], whereas that of Pt/SrTiO₃ was 35 $\mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$ [61]. For Mo-doped ZnIn₂S₄, an H₂ evolution rate of 138.8 $\mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$ was achieved using lactic acid as a sacrificial agent [62]. The H₂ evolution rate of PAN was 240 $\mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$ [47], while among polymer-containing photocatalysts, Cu@g-PAN/STO exhibited an H₂ evolution rate of 265.25 $\mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$ using 10% TEOA [48].

The incorporation of Rh/Cr/Co cocatalysts into PAN/ASTO resulted in an increase in photocatalytic activity. The H₂ evolution rate reached 319.5 $\mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$, which was approximately 6.64 times higher than that of PAN/ASTO and 1.96 times higher than that of PAN/STO.

In summary, the obtained PAN/ASTO@Rh/Cr/Co exhibits promising photocatalytic activity compared with previously reported one-dimensional photocatalysts.

4. Conclusion

This study investigated the synthesis and characterization of PAN/STO, PAN/ASTO, and PAN/ASTO@Rh/Cr/Co carbon nanofiber composites using the electrospinning method followed by carbonization. SEM analysis of the morphology and elemental composition confirmed the successful formation of the composite carbon nanofibers. The results of photoelectrochemical measurements demonstrated an increase in photocurrent density for the PAN/ASTO@Rh/Cr/Co sample, indicating effective separation of photogenerated electron-hole pairs and a reduction

in their recombination rate. Photocatalytic hydrogen evolution tests showed that the PAN/ASTO@Rh/Cr/Co composite exhibited the highest hydrogen evolution rate of 319.5 $\mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$ among the studied materials.

The results demonstrate that PAN/ASTO@Rh/Cr/Co exhibits substantially higher photoelectrochemical and photocatalytic performance than PAN/ASTO and PAN/STO. Comparison of PAN/STO and PAN/ASTO shows that Al doping alone does not account for the enhanced H₂ evolution under the investigated conditions. The pronounced improvement observed for PAN/ASTO@Rh/Cr/Co is associated with the combined Rh/Cr/Co cocatalyst modification. However, because control samples containing the individual Rh, Cr, and Co components were not investigated, their separate contributions to the overall photocatalytic activity cannot be determined from the present dataset.

Further research will focus on evaluating the photocatalytic stability of the synthesized composite nanofibers, determining the optimal conditions for photocatalytic hydrogen production by varying the parameters of the reaction medium, and systematically investigating the individual roles of Rh-, Cr-, and Co-containing cocatalysts using appropriate control samples.

CRedit authorship contribution statement

Saule Dyussembekova and Aigerim Serik: Conceptualization, Methodology, Writing – original draft.
Aididar Sailaubek: Visualization, Investigation. **Aibol**

Baratov: Software, Methodology. **Diana Yessenkel-dina:** Writing – Review & Editing, Resources. **Chingis Daulbayev:** Writing – review & Editing, Supervision, Conceptualization. **Ulzhan Abdikarimova:** Data Curation, Writing – Original Draft.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary material

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