

## EXPERIMENTAL STUDY ON THE EFFECTS OF LIGHT INTENSITY ON ENERGY CONVERSION EFFICIENCY OF PHOTO-THERMO CHEMICAL SYNERGETIC CATALYTIC WATER SPLITTING

by

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*Hydrogen production from water using a catalyst and solar energy was an ideal future fuel source. In this study, an elaborate experimental test rig of hydrogen production from solar water splitting was designed and established with self-controlled temperature system. The effects of light intensity on the reaction rate of hydrogen production from solar water splitting were experimentally investigated with the consideration of optical losses, reaction temperature, and photo-catalysts powder cluster. Besides, a revised expression of full-spectrum solar-to-hydrogen energy conversion efficiency with the consideration of optical losses was also put forward, which can be more accurate to evaluate the full-spectrum solar-to-hydrogen energy of photo-catalysts powders. The results indicated that optical losses of solar water splitting reactor increased with the increase of the incoming light intensity, and the hydrogen production rate increased linearly with the increase of effective light intensity even at higher light intensity region when the optical losses of solar water splitting reactor were considered.*

Key words: hydrogen, solar energy, water splitting, energy conversion efficiency

### Introduction

As the rapid growth in population, urbanization and industrialization, the energy needs of the world are increasing day by day [1]. Till now, the whole primary energy consumption of the world is mainly from fossil fuel based energy sources [2-4]. The utilizations of fossil fuel induce a series of environmental issues, such as: acid rain, ozone depletion, and global warming [5, 6]. Hydrogen is considered to be one of the most effective solutions of environmental problems, which can be storable with high energy density [7, 8]. However, most of the hydrogen for commercial application is produced from fossil fuel reforming and water electrolysis, in which a large amount of fossil fuel is combusted or electric energy is consumed [9-12]. Therefore, seeking for cost effective and sustainable strategies to produce hydrogen from renewable energy resources is an urgent task [13,14].

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Taking advantage of the abundance in solar energy for hydrogen production without any green house gas emissions and pollutions has attracted strong interests [15-18]. Photocatalytic water splitting in to  $H_2$  and  $O_2$  using semiconducting catalysts has attracted much interests due to the potential of this technology, as well as the great economic and environmental interest for the production of the clean fuel  $H_2$  from water using solar energy [19]. Photocatalytic water splitting to produce hydrogen using semiconductor materials as photo-catalyst dates back to 1972 when Fujishima and Honda demonstrated the potential of  $TiO_2$  semiconductor materials to split water into  $H_2$  and  $O_2$  [20]. During the past years, a large amount of photo-catalysts had been developed to split water into  $H_2$  and  $O_2$  with relative high energy conversion efficiencies under UV and visible light illumination [21].

Although many photocatalytic systems based on metal oxides had been developed for the water splitting, most of them required ultra violet light due to the large band gap of these semiconductor materials [22, 23]. As nearly half of the solar energy incident to the Earth's surface lied in the visible spectrum region, it was essential to use visible light efficiently to produce  $H_2$  on a large scale by photocatalytic water splitting. Maximum solar conversion efficiency for photocatalytic water splitting with a quantum efficiency of 100% can be calculated using the standard solar spectrum. Even if all ultra violet light up to 400 nm can be utilized, the full-spectrum solar conversion efficiency would be only up to 2%, which was similar to the maximum conversion efficiencies of photosynthesis in green plants under normal environmental conditions (1-2%) [24]. Therefore, the full-spectrum solar-to-hydrogen energy conversion efficiency (ECE) was more suitable to be used to evaluate the effective utilization of solar energy during the photocatalytic water splitting process. The conventional definition of full-spectrum solar-to-hydrogen ECE for hydrogen production from solar water splitting  $E_c$  was expressed [25]:

$$E_c = \frac{\Delta G_{H_2}^0 R_{H_2}}{I_T A} \quad (1)$$

where  $\Delta G_{H_2}^0$  [ $Jmol^{-1}$ ] denoted the standard Gibbs energy for the chemical reaction forming  $H_2$ ,  $R_{H_2}$  [ $mols^{-1}$ ] denoted the rate at which the products were formed,  $I_T$  [ $Wm^{-2}$ ] was the total incoming light irradiance, and the symbol  $A$  [ $m^2$ ] was the irradiated area.

The researchers had developed a fully integrated system of nanoscale photo-electrodes assembled from inorganic nanowires for direct solar water splitting, and the artificial photosynthetic system comprised two semiconductor light absorbers with large surface area, an interfacial layer for charge transport, and spatially separated co-catalysts to facilitate the water reduction and oxidation. Under simulated sunlight, a 0.12% full-spectrum solar-to-fuel conversion efficiency was achieved, which can be comparable to that of natural photosynthesis [26]. The full-spectrum solar-to-hydrogen ECE of at least 0.8% had been obtained in the hydrogen production from solar water splitting by Houlihan *et al.* [27], where  $TiO_2$  semiconductor electrodes were prepared by heat-treatment of titanium metal foils. The experimental results obtained by Houlihan *et al.* [27] indicated the possibility of utilizing a greater portion of the solar spectrum, thereby increasing overall conversion efficiency even further. The  $Cd_{1-x}Zn_xS$  solid solution with nanotwin structures were synthesized by Liu *et al.* [28] for photo catalysis of water splitting, and the twins in  $Cd_{1-x}Zn_xS$  solid solution had exhibit superior photocatalytic activities under visible light irradiation (430 nm) without noble metal co-catalysts, the ECE under direct solar irradiation (with infrared light filtered by water) was as high as 6.0%

As known, the reaction rate of photocatalytic water splitting was not only highly dependent on the photo catalysts, but also dependent on test conditions, such as reaction temperature and light intensity [29-31]. In fact, the effects of reaction temperature on the reaction rate of photocatalytic water splitting still had controversial arguments and cannot reach a consensus. Liu *et al.* [32] regarded that the increase of reaction temperature can induce the increase of energy losses of photo-excited electrons and positive holes from the standpoint view of thermodynamics for a single electron-hole pair, and the increase of reaction temperature would be negative to the photo catalysts in theory. However, the experimental results conducted by Ritterskamp *et al.* [33] indicated that higher hydrogen production efficiencies can be obtained with the increase of reaction temperature, where titanium disilicide ( $\text{TiSi}_2$ ) was adopted as photo catalyst for water splitting.

Besides temperature, the effects of light intensity on the reaction rate of photocatalytic water splitting also had controversial viewpoints. Domen [34] was the pioneer to investigate the light intensity dependence in photocatalytic decomposition of water over  $\text{K}_4\text{Nb}_6\text{O}_{17}$ . Their experimental results indicated that the reaction rate of photocatalytic water splitting increased linearly with light intensity in the lower light intensity region. Sacrificial hydrogen productions from formaldehyde with Pt- $\text{TiO}_2$  photo catalyst under different solar light intensities were measured by Chowdhury *et al.* [35], and the light intensity of simulated sunlight varied in the range of  $300 \text{ W/cm}^2$  to  $1000 \text{ W/cm}^2$ . Their experimental results indicated that the reaction rate of photocatalytic water splitting followed power-law dependence on light intensity. However, the latest experimental test conducted by Liu *et al.* [36] indicated that the photo catalysis reaction rate grew exponential with light intensity, fig. 1. It should be noted the light intensity was very low and varied in the range of  $34.2 \text{ W/cm}^2$  to  $47.4 \text{ mW/cm}^2$  with only three tested points [36].

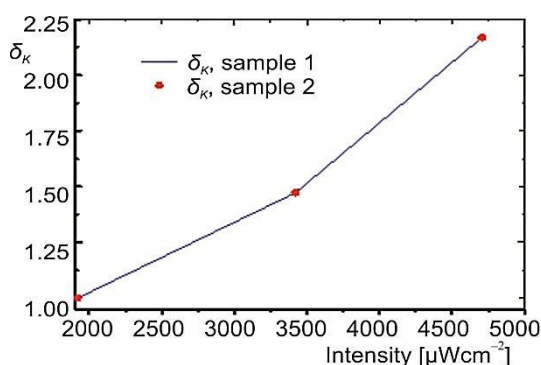
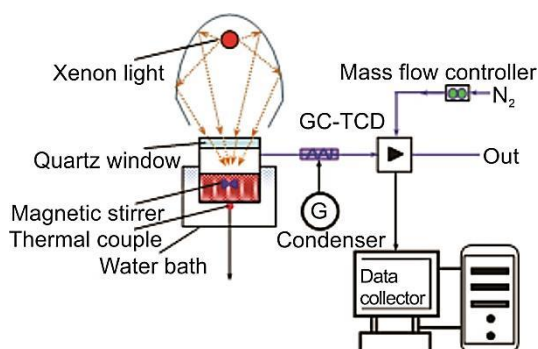


Figure 1. Effects of radiation intensity on photo catalysis tested by Liu *et al.* [36]

Based on the previous literature survey, it can be concluded that the effects of light intensity on the reaction rate of hydrogen production from solar water splitting still had controversial viewpoints. Besides, the effects of optical losses were not considered in the previous studies when the effects of light intensity on the reaction rate of photocatalytic water splitting were investigated. As illustrated previously, the reaction rate of hydrogen production from solar water splitting was influenced by several factors, such as: light intensity, reaction temperature, particle size, and rotation rate of magnetic stirring. In this study, an elaborate experimental test rig was designed and established with self-controlled temperature system and ultrasonic system, which can test one influence factor while the other influence factors can be kept constant. The effects of light intensity on the reaction rate of hydrogen production from solar water splitting were experimentally investigated with the consideration of optical losses. A fitting expression of hydrogen production rate with the change of light intensity was put forward with the consideration of optical losses. Besides, the revised expression of full-spectrum solar-to-hydrogen ECE with the consideration of optical losses was also put forward in this study.

### Test rig of hydrogen production from solar water splitting reaction

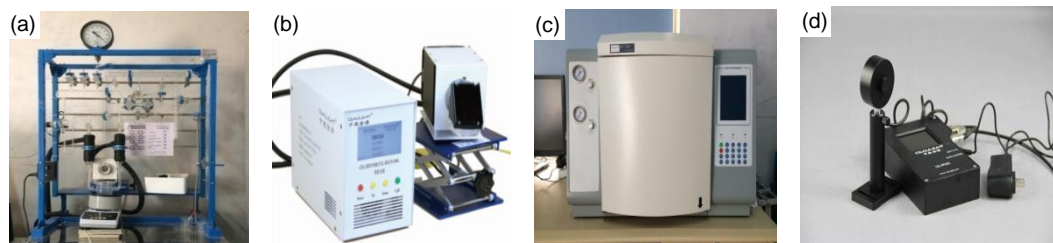
In this study, an elaborate hydrogen production for solar water splitting reaction test rig was designed and established. The schematic of photocatalytic water splitting reactor placed in a constant-temperature water bath which was irradiated by Xenon light source was shown in fig. 2. A glass photocatalytic water splitting reactor with a high transmissivity quartz cover was used to position the samples. A constant-temperature water bath system was designed to accurately control the temperature of photocatalytic water splitting reactor (55 °C in this study). Solutions with photo catalyst powders were dispersed by a magnetic stirrer in an aqueous (100 mL) containing ethylene glycol, (CH<sub>2</sub>OH)<sub>2</sub>, 0.02 mL, as electron donors. A magnetic stirrer with rotation speed display was adopted to disperse the photo catalyst powders, which can guarantee the rotation speed (700 rpm) to be the same for each test case.



**Figure 2. Schematic of photocatalytic water splitting reactor placed in a constant-temperature water bath which was irradiated by Xenon light**

(CEAULIGHT, CEL-HXF300). The spectral range of the Xenon light source was 300~2500 nm. The output power of ultraviolet light (<390 nm) of the Xenon light source was 2.6 W and the output power of infrared light (>770 nm) of the Xenon light source was 28.8 W.

A 0.5 wt.%-Pt co-catalyst for the promotion of hydrogen evolution was photodeposited *in situ* on the photo catalyst TiO<sub>2</sub> (Degussa-P25, average particle size of 21 nm) from the precursor of H<sub>2</sub>PtCl<sub>6</sub>·6H<sub>2</sub>O. The amount of H<sub>2</sub> gas was detected using online gas chromatograph equipped with a thermal conductivity detector (GC-TCD) while N<sub>2</sub> was purged as carrier (CEAULIGHT, GC7920). An intense-light power-density meter (CEAULIGHT, CEL-NP2000-2) was used to monitor the light irradiance from the Xenon light source. The spectral response range of power-density meter is 0.19-2.5 μm with uniformity of 2.7%, and the resolution of power-density meter was 0.1 μW with less than 5% measurement errors. Before inhaling H<sub>2</sub> to the GC-TCD, a condenser was adopted to condense the vapor pipeline to avoid damaging the GC-TCD by steam. Photographs of the hydrogen production for solar water splitting reaction test rig set up in Harbin Institute of Technology were presented in fig. 3.



**Figure 3. Photographs of the hydrogen production for solar water splitting reaction test rig set-up in Harbin Institute of Technology; (a) Reactor, (b) Xenon light source, (c) GC-TCD, and (d) Power-density meter**

### Characterization of photo-catalyst samples

Figure 4 presented the TEM and HRTEM images of the obtained Pt/TiO<sub>2</sub> nanoparticles. As shown in this figure, the average diameter of the nanoparticles was ~21 nm, and Pt nanoparticles with average diameter of ca. 10-25 nm were observed on the surface of TiO<sub>2</sub> nanoparticles. Figure 4 also exhibited the HRTEM image of the nanoparticles, the clear lattice fringe with a spacing of 0.428 nm and 0.237 nm can be matched well with the (002) and (112) plane of tetragonal TiO<sub>2</sub>, respectively. Meanwhile, the fringe spacing of 0.196 nm matched well with the (200) plane of Pt.

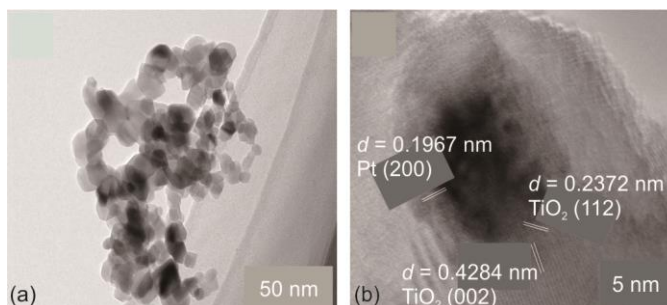


Figure 4. The TEM and HRTEM images of the obtained photo-catalyst nanoparticles

### Results and discussion

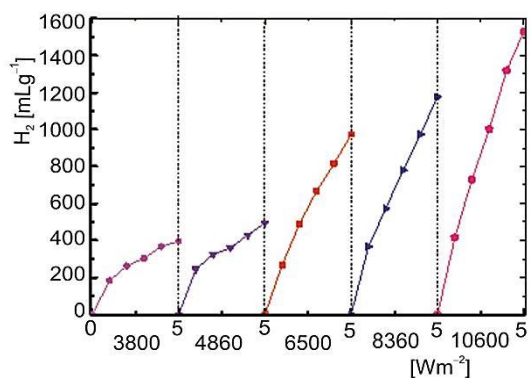
#### Effects of light intensity on hydrogen production

In this study, the effects of light intensity on hydrogen production from solar water splitting reaction were investigated while the photo-catalysts powders were oscillated by ultrasonic before test to eliminate the cluster of photo catalyst powders. The photo catalyst powders (0.01 g) were immersed in 3 mL water and oscillated two minutes every two minutes for three times by ultrasonic wave to prevent the cluster of photo catalyst powders before solar water splitting reaction. The variation of light intensity was controlled by changing the current of the Xenon light source. Five cases were selected, 10 A, 12.5 A, 15 A, 17.5 A, and 20 A. The light intensity on the top surface of solar water splitting reactor was monitored by power-density meter. Besides, the optical losses on the bottom surface and side surfaces of solar water splitting reactor were also monitored as the solutions in the reactor cannot totally absorb the incoming sunlight. The light intensities on the top surface, bottom surface and side surfaces of the solar water splitting reactor under different currents of the Xenon light source were listed in tab. 1. As seen in this paper, the optical losses increased with the increase of the incoming light intensity, and the optical losses of the solar water splitting reactor need to be considered.

Table 1. Light intensities on the surfaces of solar water splitting reactor under different currents of the Xenon light source

Current, A	10	12.5	15	17.5	20
$I_T$ [Wm <sup>-2</sup> ]	3800	4860	6500	8360	10600
$I_B$ [Wm <sup>-2</sup> ]	164	217	281	393	460
$I_{S-L}$ [Wm <sup>-2</sup> ]	41.8	46.2	63.7	91.2	114.5
$I_{S-R}$ [Wm <sup>-2</sup> ]	40.6	52.4	71.6	100.9	130.9
$I_E$ [Wm <sup>-2</sup> ]	3368.8	4323.2	5780.2	7344.0	9344.1

Figure 5 presented the time courses of H<sub>2</sub> evolution catalyzed by photo-catalysts powders with different incoming light intensities under 300 W Xenon light source irradiation. As shown in this figure, the light intensity variation had significant impacts on H<sub>2</sub> production



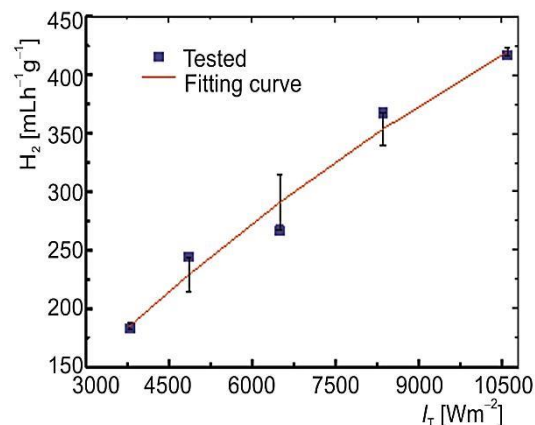
**Figure 5. Time courses of H<sub>2</sub> evolution catalyzed by photo-catalysts powders with different incoming light intensities**

was easy to cluster at a long time of magnetic stirring, the hydrogen production at the following hours which was lower than that in the first hour was induced due to the reason that the cluster of photo-catalysts powders in the following hours.

In order to obtain a more accurate fitting expression between hydrogen production rate and light intensity with the elimination of the clusters of photo-catalysts powders, the hydrogen production in the first hour under different light intensity was adopted. The hydrogen production rate variation with the increase of light intensity without the consideration of optical losses was shown in fig. 6. As seen in this figure, the hydrogen production rate did not increase linearly with the increase of light intensity. A fitting expression between hydrogen production rate and light intensity with maximum relative error 8.98% was put forward:

$$M_{H_2} = 835.016 \left[ 1 - \exp(-6.59013 \cdot 10^{-5} I_T) \right] \quad (2)$$

As seen from the above equation, hydrogen production rate varied exponentially with the increase of light intensity when the optical losses were not considered.



**Figure 6. The H<sub>2</sub> production rate variation with the increase of light intensity without the consideration of optical losses**

during the solar water splitting reaction. The total amount of H<sub>2</sub> production in the tested five hours increased with the increase of light intensity. Figure 5 also presented that the H<sub>2</sub> production rate in the first hour was much higher than that in the following hours. For example, the H<sub>2</sub> production rate was 367.83 mL/h/g in the first hour when the light intensity was 8360 W/m<sup>2</sup>, while it decreased to 205.24 mL/h/g, 207.19 mL/h/g, 193.70 mL/h/g and 202.31 mL/h/g in the following four hours, respectively.

This phenomenon can be explained as follows: the photo-catalysts powders (average particle diameter was 21 nm) was a kind of colloidal dispersion (1~100 nm) which

As known, the light intensity used in this study was much higher than the maximum light intensity (1000 W/m<sup>2</sup>) adopted by Domen [34] and Chowdhury *et al.* [35]. The experimental results obtained in this study shared the same conclusion with those obtained by Domen [34] and Chowdhury *et al.* [35] that the hydrogen production rate did not vary linearly with the increase of light intensity at higher light intensity region.

However, these fitting expressions did not consider the effects of optical losses of the solar water splitting reactor. The used light intensities in the above analysis as well as Domen [34] and Chowdhury *et al.* [35] were the values on the top surfaces of reac-

tor, but not the values absorbed by the solution containing photo-catalysts powders. Therefore, an effective light intensity was put forward in this study to accurately express the light intensity absorbed by the solutions. The definition of effective light intensity was expressed:

$$I_E = \frac{I_T A_T - I_B A_B - 0.5(I_{S-L} + I_{S-R}) A_S}{A_T} \quad (3)$$

where the symbols  $I_T$ ,  $I_B$ ,  $I_{S-L}$ , and  $I_{S-R}$  delegated the light intensities on the top surface, bottom surface, left side surface, and right side surface of the solar water splitting reactor, respectively. The variations of effective light intensity with the increase of the current of Xenon light source were also listed in tab. 1.

The hydrogen production rate variation with the change of effective light intensity was shown in fig. 7, which had considered the optical losses of the solar water splitting reactor. As seen in this figure, the hydrogen production rate increased almost linearly with the increase of effective light intensity at higher light intensity region. A revised fitting expression between hydrogen production rate and effective light intensity with maximum relative error 7.08% was put forward:

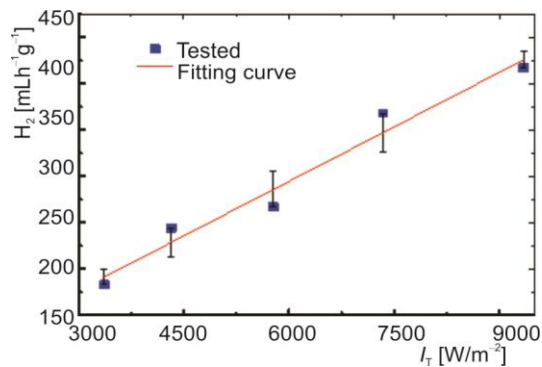


Figure 7. The  $H_2$  production rate variation with the increase of effective light intensity

$$M_{H_2} = 59.20238 + 0.03922I_E \quad (4)$$

Based on the previous studies, it can be concluded that the previous expression of hydrogen production rate variation with light intensity deviated from the intrinsically tendency if the optical losses of solar water splitting reactor were not considered.

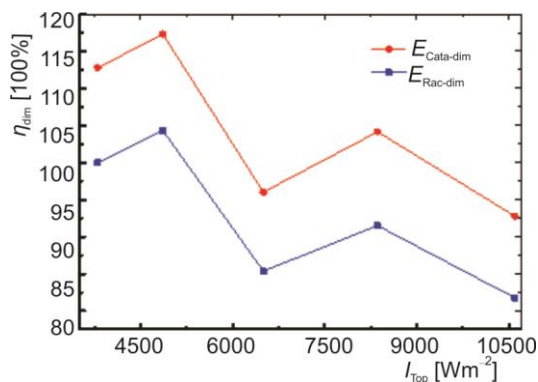
#### Full-spectrum solar-to-hydrogen conversion efficiency analysis

As illustrated in eq. (1) previously, the conventional definition of full-spectrum solar-to-hydrogen ECE for hydrogen production from solar water splitting omitted the optical losses of reactor. Therefore, an improved definition of full-spectrum solar-to-hydrogen ECE for solutions containing photo-catalysts powders,  $E_{cata}$ , was put forward as eq. (2), which can be more accurate to evaluate the full-spectrum solar-to-hydrogen conversion efficiency of photo-catalysts powders:

$$E_{cata} = \frac{\Delta G_{H_2}^0 R_{H_2}}{I_T A_T - I_B A_B - 0.5(I_{S-L} + I_{S-R}) A_S} \quad (5)$$

where, the term  $I_T A_T$  delegated the total incoming solar energy, and the term  $I_B A_B$  denoted the optical losses from the bottom surface of reactor, and the term  $0.5(I_{S-L} + I_{S-R}) A_S$  denoted the optical losses from the side surface of reactor.

Although the production rate in the unit of mL/h/g was high, the amount of total hydrogen production was relative low due to the photo-catalysts powders used in this study was very low (0.01 g). Therefore, the dimensionless full-spectrum solar-to-hydrogen ECE,  $E_{dim} = E/E_{\text{reac-10A}}$  was adopted to analyze the effects of optical losses on solar-to-hydrogen ECE. Figure 8 presented the variation of dimensionless full-spectrum solar-to-hydrogen ECE with



**Figure 8. Variations of dimensionless full-spectrum solar-to-hydrogen energy conversion efficiency**

was 81.78%, whereas the real dimensionless full-spectrum solar-to-hydrogen ECE for solutions containing photo-catalysts powders was 92.77%.

## Conclusions

An elaborate experimental test rig was designed and established with self-controlled temperature system and ultrasonic system. The effects of light intensity on the reaction rate of hydrogen production from solar water splitting were experimentally investigated with the consideration of optical losses, reaction temperature and photo-catalysts powder cluster. Besides, a revised expression of full-spectrum solar-to-hydrogen ECE with the consideration of optical losses was also put forward, which can be more accurate to evaluate the full-spectrum solar-to-hydrogen energy of photo-catalysts powders. The following conclusions can be drawn as follows.

- Hydrogen production rate grew exponential with the increase of the incoming light intensity at higher light intensity region when the optical losses of solar water splitting reactor were not considered.
- When the optical losses of solar water splitting reactor were considered, hydrogen production rate increased linearly with the increase of effective light intensity even at high light intensity region.
- The full-spectrum solar-to-hydrogen ECE of solar water splitting reactor was at least 10% lower than that of photo-catalysts powders.

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different light intensities under 300 W Xenon light source irradiation. As seen in this figure, the whole tendency of solar-to-hydrogen ECE decreased with the increase of light intensity. For example, the dimensionless full-spectrum solar-to-hydrogen ECE with the consideration of optical losses under  $I_T = 10600 \text{ W/m}^2$  was 10% lower than that under  $I_T = 3800 \text{ W/m}^2$ . Besides, the dimensionless full-spectrum solar-to-hydrogen ECE was at least 10% lower than that with the consideration of optical losses. Take  $I_T = 10600 \text{ W/m}^2$  as an example, the dimensionless full-spectrum solar-to-hydrogen ECE without the consideration of optical losses



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