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Synthesis of SnO_2 and TiO_2 Semiconductor Quantum Dots for Hydrogen Gas Evolution through Water Splitting Reaction

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ABSTRACT

This paper reports on the synthesis, characterization of tin dioxide (SnO_2) as well as titanium dioxide (TiO_2) quantum dots (QDs) and compare their potential production of hydrogen gas (H_2) through photocatalytic water splitting reaction. One pot synthesis of SnO_2 QDs was prepared through thermal pyrolysis of tin stearate under non-hydrolytic approach. On the other hand, synthesis of TiO_2 QDs was prepared through the mild condition of $TiCl_4$ with the use of icy bath. According to HRTEM image, the shape of SnO_2 QDs is almost quasi-spherical and TiO_2 QDs is almost spherical in shape. Both nanoparticles have diameter ranged in 1-10 nm, while XRD patterns are matched well with standard pattern of SnO_2 and TiO_2 QDs, respectively. Optical studies reveal that both SnO_2 and TiO_2 are of UV-active compounds and hence, can be selected as the candidates for hydrogen production through UV activated photocatalytic water splitting reaction.

Keywords: Tin dioxide; titanium dioxide; optical properties; quantum dots; photocatalyst

INTRODUCTION

Solar radiation has the high potential to be a renewable energy source which is environmentally clean and economically energy. However, there is a severe challenge to convert and store solar energy efficiently. Photocatalytic H_2 production by water splitting is one of the alternatives, but only low conversion efficiencies have been reported up to now [1]. The efficiency of photocatalytic ability, particularly for hydrogen production through water splitting reaction are depends on three factors: efficiency of optical

absorption by the photocatalyst, efficiency of creation of charger carriers, and efficiency of use of the charge carriers in the photocatalytic processes.

Photocatalysis including electron-hole pair formation proposed by band-gap excitation of a semiconductor particle. When a photon with energy ($h\nu$) matches or exceeds the band-gap energy of the semiconductor, an electron (e^-) is moved from the valence band into the conduction band, leaving a hole (h^+) behind. The substance that reacts with electron and hole must suitable, if not the saved energy is decline within a few nanoseconds by recombination. If the electrons and holes move without recombination to the surface of the semiconductor, they can participate in various oxidation and reduction reactions with adsorbed species such as water, oxygen, and other organic or inorganic species. However, there are possibility that electron in conduction band unexcited and react with adsorbed species, then it recombines back with the hole in valence band. Sacrificial agent is needed to overcome this issue to promote the photocatalyst's efficiency.

There are many candidate semiconductors that can be used as photocatalysts in water splitting such as SnO_2, ZnO, ZrO_2, $SrTiO_3$, CdS, MoS_2, Fe_2O_3, and WO_3. Among these semiconductors, TiO_2 nanomaterials are the most studied semiconductor. There are good reasons for comparing SnO_2 with TiO_2 nanomaterials because TiO_2 was proved to be an efficient, biologically and chemically inert, inexpensive, resistant to photocorrosion and chemical corrosion, highly photoactive, and was also proven to have a potential to completely oxidize a variety of organic compounds, including persistent organic pollutants [2]. There are two phases of TiO_2 photocatalyst: anatase and rutile. Each titanium ion is at the center of an oxygen octahedron in both structures. Difference between these two phases are he oxygen ions form a slightly distorted hexagonal compact lattice in rutile whereas the oxygen forms a cubic face centered (cfc) crystallographic lattice in anatase.

The main focus in this study is to investigate the effectiveness of quantum dots semiconductors to be used as photocatalyst in water splitting reaction to produce H_2 gas. As such, two types of quantum dots are of interest namely SnO_2 QDs and TiO_2 QDs. Pyrolysis method has been used to produce SnO_2 QD and TiO_2 QDs has been prepared by using $TiCl_4$ precursors via mild condition. The two quantum dots then characterized by using different analytical techniques to elucidate the crystallinity, morphology and optical properties. Photocatalytic water splitting hydrogen measurement is then conducted to the samples and then being compared in term of the H_2 production rate.

MATERIALS AND METHODS

Synthesis of tin dioxide (SnO_2) quantum dots

A total of 1.0 mmol of stannum stearate ($C_{36}H_{70}O_4Sn$, Meryer Chemicals), 0.75 mmol of oleic acid ($C_{18}H_{34}O_2$, Sigma-Aldrich, 90%), 25 mL of n-hexadecane ($C_{16}H_{34}$, Merck, 99%), 0.75 mmol of oleylamine, and 0.75 mmol of 1,2-dodecanediol was loaded into a 250 mL four neck flask equipped with a Allihn condenser. The mixture is then heated by using a heating mantle and the temperature is regulated by a temperature controller (JKEM Reaction Controller-SYR-DTC) under vigorous stirring. The mixture being heated to 120 ^oC and hold for 1 h at first. After 1 h, the temperature was increased to 287 ^oC under constant heating rate and the mixture has been refluxed for 5 h. For

chronological observation, the mixture was withdrawn for every 1 h. After the reaction completed, SnO_2 QDs collected by using 2 mL of hexane (solvent) and 5 mL acetone (non-solvents) through a series of centrifugation and resuspension process. Finally, the yellow precipitate was dried at 80 ºC in the oven.

Synthesis of titanium dioxide (TiO_2) quantum dots

Firstly, titanium tetrachloride ($TiCl_4$, Merck Co.) was dissolved into distilled water in an icy bath under vigorous agitation to obtain a 50 wt.% of $TiCl_4$ homogeneous aqueous solution. Separately, 4 mL of tetrabutyl titanate (TBT, Sigma Aldrich) was added into 30 mL of toluene in an icy condition at a reaction flask. Subsequently, 4 mL of $TiCl_4$ aqueous solution was added dropwise into TBT/toluene solution under magnetic stirring. The mixture then keep for continuous stirring for 1 h until white precipitate is observable, the product was collected and alternatively wash with ethanol and distilled water to obtain the TiO_2 QDs. The product will be dried in a hot air oven at 70-75 °C for 2 h.

Characterizations

The dried products of SnO_2 and TiO_2 were analyzed by XRD (PANalytical EMPYREAN X-ray diffractometer), Cu Ka irradiation (40 kV/35 mA) to study the crystallinity of SnO_2 and TiO_2. The morphology and lattice spacing will be evaluated by high resolution transmission electron Microscopy (HRTEM, JEM-2100F) with operational voltage of 200 kV. Before the HRTEM imaging, SnO_2 and TiO_2 quantum dots need to be diluted with ethanol and drop-casted on 300 mesh carbon-coated copper grid. UV-visible absorption spectra of synthesized SnO_2 QDs and TiO_2 QDs were respectively recorded with Perkin-Elmer Lambda 750 UV-VIS-NIR spectrophotometer from 200-800 nm.

Photocatalytic water splitting reaction

Hydrogen gas (H_2) production through water splitting reaction was conducted in quartz-made photocell. A 2.80 mg of the as-synthesized SnO_2 QDs was loaded into photocell that contained 3.8 mL water and 0.42 mL ethanol. The mixture was irradiated with Xenon arc lamp (Newport 69911) with output intensity of 100 mW/cm^2. The hydrogen gas evolved was measured by using Agilent 7820A gas chromatography system to analyze the hydrogen generation over the time. Similarly, the hydrogen gas production through photocatalyzed of TiO_2 quantum dots will be repeated using the same observation of time and the result is then collected. The efficiency of H_2 gas production through two different quantum dots photocatalyst were compared and evaluated.

RESULTS AND DISCUSSION

TEM analysis

Figures 1(a) and (b) present the HRTEM images of the as-prepared SnO_2 and TiO_2 quantum dots, respectively. It can be observed that the particles exhibited almost quasi-spherical in shape for SnO_2 and almost spherical for TiO_2 QDs. The mean particle size of synthesized SnO_2 quantum dots was in the range of 2-11 nm. The magnified HRTEM of single particles presented is having a diameter of ~6 nm. The calculated interplanar *d*-spacing values are to be 3.075 Å and 3.270 Å which can be due to the plane of (110) of *hkl* miller indices. On the other hand, the average particle size of TiO_2 quantum dots

viewed to be ranged from 1-9 nm, which is much smaller than SnO_2 quantum dots. Possible explanation can be due to the mild synthesis condition involved during the preparation of TiO_2 under icy bath. The high magnification image of single particle of TiO_2 quantum dot revealed that the synthesized particle is of ~5 nm. Detailed interplanar spacing value has been determined to be 2.67 Å and this value is due to the (130) plane. These results confirmed the synthesis of TiO_2 and SnO_2 quantum dots have been successfully prepared.

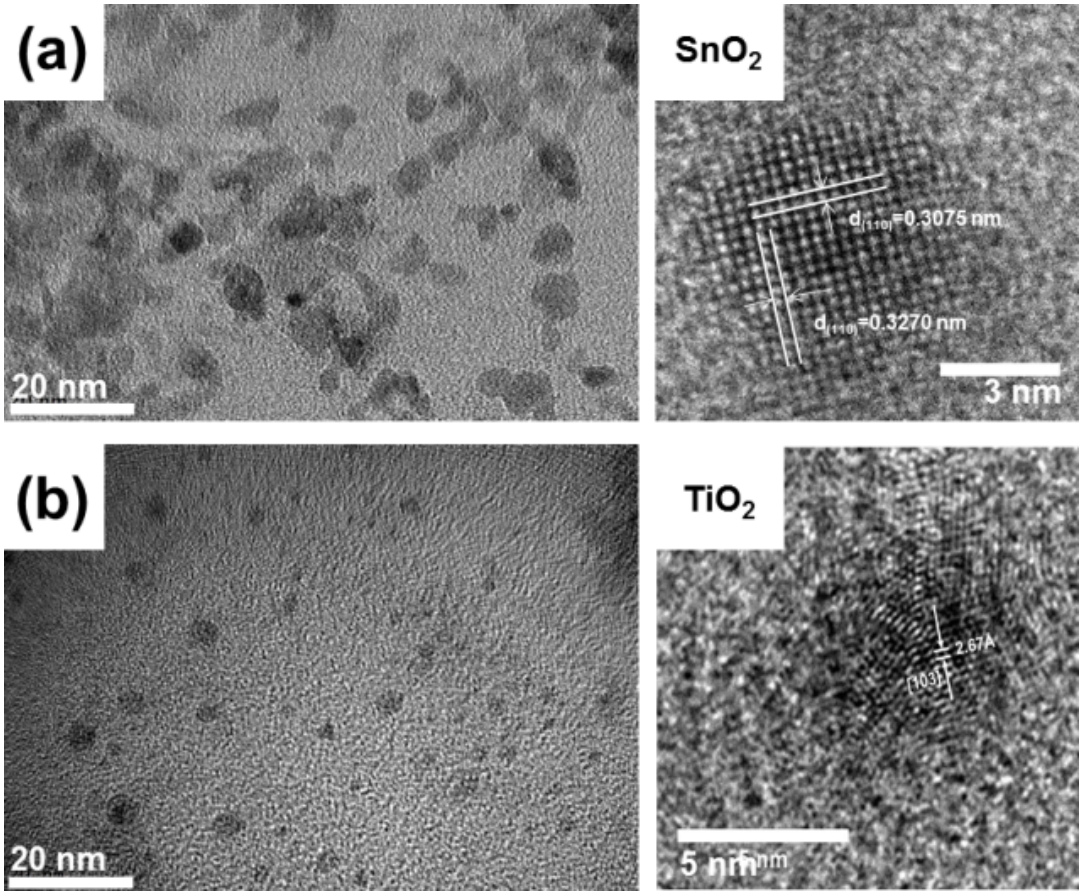


Figure 1. HRTEM images of (a) SnO_2 quantum dots and (b) TiO_2 quantum dots

XRD patterns analysis

The XRD patterns of SnO_2 quantum dots and TiO_2 quantum dots are shown in Figure 2(a) and (b), respectively. As observed from Figure 2(a), all the diffraction peaks are well-indexed to the rutile phase of SnO_2 (JCPDS file no: 00-041-1445) without the presence of other mixture of phases. The XRD pattern for TiO_2 quantum dots is presented in Figure 2(b). From the XRD patterns of TiO_2 QDs, the dominant peaks are well-matched with the standard reference pattern (JCPDS reference no: 00-021-1272) indicated the presence of anatase phase of TiO_2. The broadening peaks of TiO_2 particle can be explained due to the nanosized of the particles are being produced [3]. All these results are consistent with the TEM characterization shown in Figure 1.

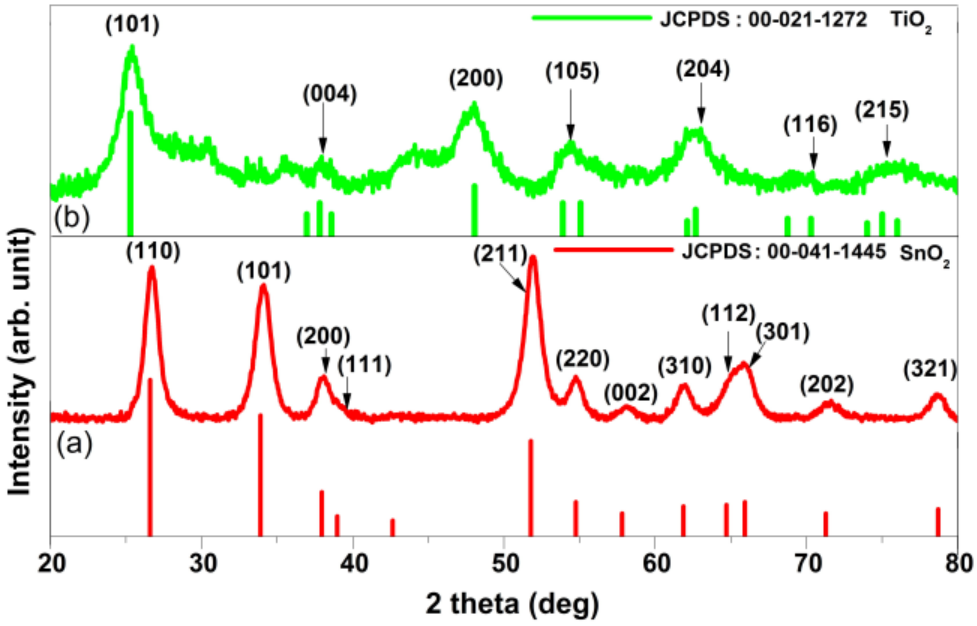


Figure 2. XRD patterns of (a) SnO_2 quantum dots and (b) TiO_2 quantum dots. Bars at bottom of (a) and (b) represent the standard crystal data of the respective samples

Optical properties

As displayed in the absorption spectra (Figure 3), both the SnO_2 quantum dots and TiO_2 quantum dots exhibited absorption edge at the range of 300-400 nm, which can be primarily ascribed to electrons promotion from the valence band to the conduction band of the samples at UV region [4]. Both the samples showed a cut-off wavelength near the onset at ~380 nm. For SnO_2 quantum dots (red line), the sample has a cut-off wavelength that extrapolated to the x-axis at 392 nm and for the TiO_2 quantum dots (green line), the cut-off wavelength at the x-axis is 375 nm. The band gap energy (Eg) for both samples can be estimated through the following equation:

$$E_g = \frac{hc}{\lambda}$$

where h is the Planck constant = 6.626×10^{-34} J s, c is speed of light = 3.0×10^{8} m s^{-1}, λ is cut-off wavelength. The calculated band gap energy for the samples are 3.17 eV for SnO_2 quantum dots and 3.32 eV for TiO_2 quantum dots, respectively. Both the samples showed quantum confinement effect due to the quantum sized of nanoparticles has been produced [5]. These results proved that the SnO_2 and TiO_2 quantum dots samples are both suitable to be used as UV-active photocatalysts in water splitting reaction to produce H_2 gas.

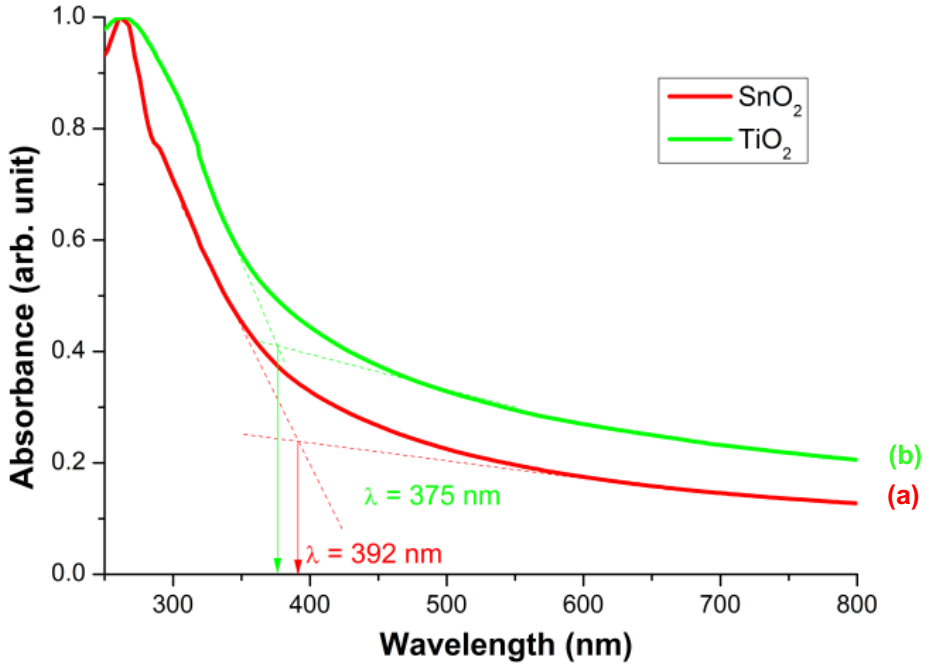


Figure 3. The UV absorbance spectra and the estimated cut-off wavelength for (a) SnO_2 quantum dots and (b) TiO_2 quantum dots

Photocatalytic water splitting hydrogen production

Hydrogen (H_2) evolution from the aqueous suspensions of SnO_2 quantum dots and TiO_2 quantum dots has been evaluated and the results were represented in Figure 4. Both of the samples were being compared with their respective commercial sample by having the same experimental condition such as constant illumination time with UV source, the addition of sacrificial agents and the quantity being used for each set of the experiment.

Figure 4 depicts the H_2 gas production for the sample of SnO_2 quantum dots that being compared with SnO_2 commercial which contained the addition of equimolar of 0.3 M Na_2S/Na_2SO_3 as sacrificial agent under 3.5 h of UV irradiation. It has been clearly evidenced that the higher H_2 production activity was obtained with SnO_2 quantum dots added aqueous suspensions with the presence of S^{2-}/SO_3^{2-} sacrificial agents. Under the constant UV illumination, the hydrogen production of SnO_2 quantum dots achieved 0.71 µmol g_{cat}^{-1} at 210 min of UV irradiation. For SnO_2 commercial suspension, the H_2 gas production achievable is 0.602 µmol g_{cat}^{-1} at 210 min of UV irradiation. The results clearly indicate that SnO_2 quantum dots suspension gives the higher H_2 evolution compared to that of SnO_2 commercial sample.

Similarly, the H_2 evolution by TiO_2 quantum dots and TiO_2 commercial added suspension with the presence of sacrificial agents has been studied and the results is represented in Figure 5. As seen in the figure, TiO_2 quantum dots (1.710 µmol g_{cat}^{-1}) added suspension has the higher H_2 production rate than that of TiO_2 commercial sample 0.591 µmol g_{cat}^{-1}, which is almost three times greater than the TiO_2 anatase commercial sample.

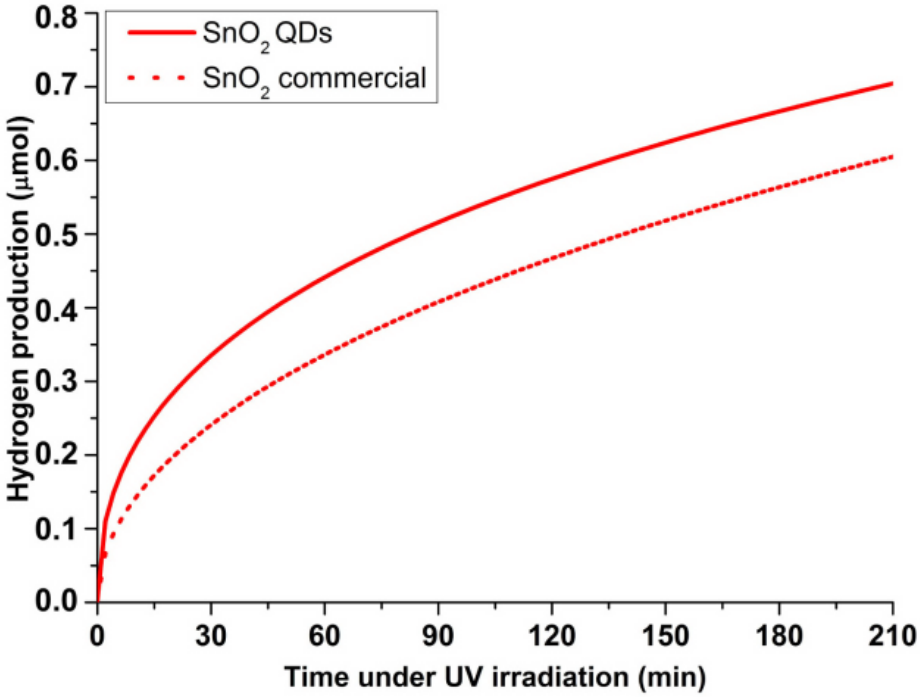


Figure 4. Hydrogen production curves for SnO_2 quantum dots (red solid line) and SnO_2 commercial (red dotted line) under 210 min of UV irradiation

All these results indicated that quantum dot samples are of the photocatalyst that gives higher H_2 production compared to the commercial products. This could be attributed to the small nanoparticle size that provide more surface-area per volume. The smaller the size of particles, the greater the surface area per volume and hence gives more active sites for the water molecules to be catalyzed in the presence of sacrificial agent [6]. The sacrificial agents served as the holes scavenger by producing more electrons to the holes produced as a result of photogenerated electrons being promoted from the valence band to conduction band [7].

This study aims to investigate the efficiency of H_2 production on both SnO_2 and TiO_2 quantum dots. Fig 6 shows the results of comparison between the as-synthesized SnO_2 and TiO_2 quantum dots. The results showed that TiO_2 quantum dot (green solid line) has the higher H_2 production as compared to SnO_2 quantum dot (red solid line). TiO_2 anatase quantum dots has been well recognized to be the great contribution to photocatalysis reaction as compared to other polymorphs like brookite and rutile [8]. Another possible explanation could be ascribed to the synthesis condition applied in this study. TiO_2 quantum dots are produced by water-based synthetic method with the use of $TiCl_4$ precursor in the presence of icy toluene-added tetrabutyl titanate (TBT) aqueous solutions. The TiO_2 quantum dots generally can be produced at low temperature and easily can be obtained into powder form which could easily be mixed with water and further enhanced the adsorption of water molecules to the surface of TiO_2 particles. Contrastingly, the SnO_2 quantum dot is being produced by pyrolysis process by decomposing the organometallic tin (II) stearate with elevated temperature. This non-water based synthetic method primarily can produce organic phase particles and thus it is

more unfavorable for the water splitting reaction as compared to water based synthesis method [9].

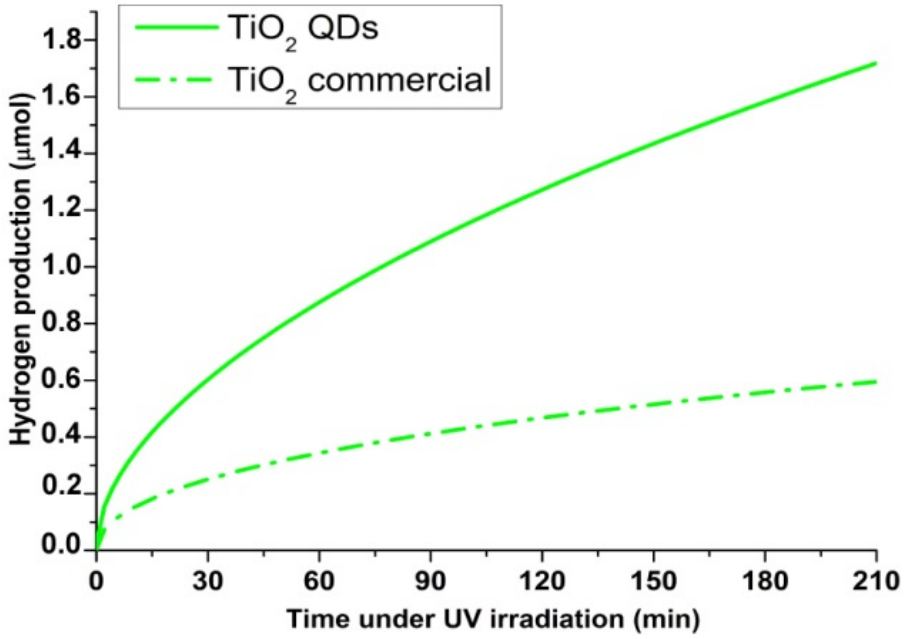


Figure 5. Hydrogen production curves for TiO_2 quantum dots (green solid line) and SnO_2 commercial (green dotted line) under 210 min of UV irradiation

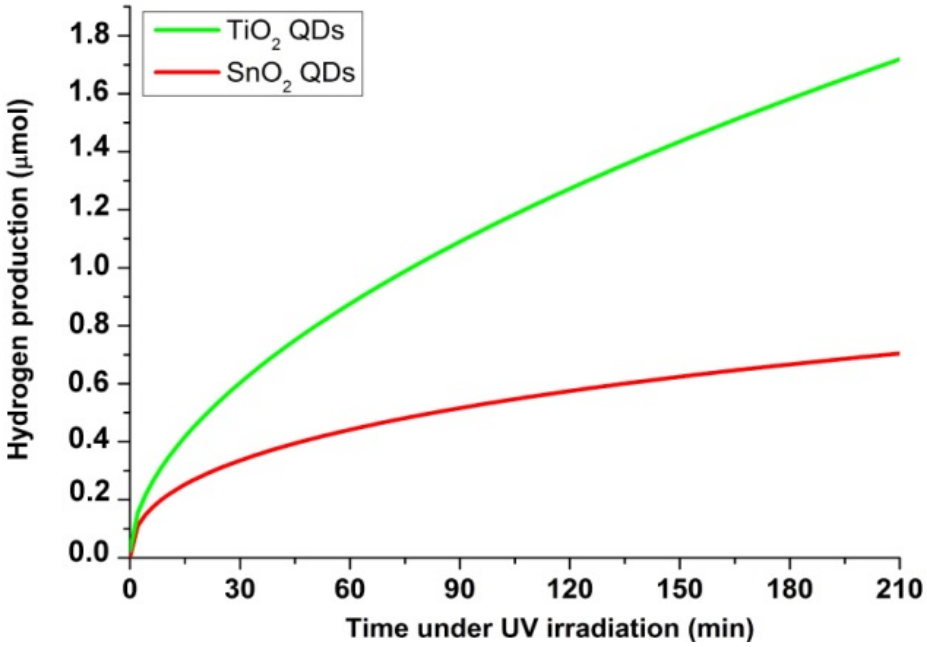


Figure 6. Comparison of H_2 production for 210 min between SnO_2 quantum dots (red solid line) and TiO_2 quantum dots (green solid line). The irradiation is held at 5 cm from the UV source

CONCLUSIONS

In summary, tin dioxide (SnO_2) quantum dots and titanium dioxide (TiO_2) quantum dots have been successfully synthesized by using two different synthetic methods. The quantum dots were characterized through HRTEM, XRD and UV-vis analytical techniques and the results suggested that both type of quantum dots are of pure SnO_2 and TiO_2 have been produced. Sacrificial agents added photocatalytic water splitting water-based suspensions for the hydrogen production indicates that TiO_2 quantum dots have the higher H_2 production over the SnO_2 quantum dots which possibly due to the crystallinity of the nanoparticle, synthetic condition, and particle size that are possibly contributing to the efficient hydrogen evolution via UV activated water splitting reaction.

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AUTHOR CONTRIBUTIONS

Investigation, experimental work, writing-original draft preparation: Ilya Mohammad Irshad Zainorin; Writing-review, investigation, validation, data curation: MeiYuen Chia; Writing-review, visualization, project administration, and funding acquisition: WeeSiong Chiu; Resources, visualization, validation: PoiSim Khiew; Software, Methodology: HongChun Lee; All authors read and approved the final manuscript.

DATA AVAILABILITY

The data supporting the findings of this study are available from the corresponding author upon reasonable request.

CONFLICT OF INTEREST

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

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