

Ph.D. Thesis

Hydrogen production using noble metal deposited TiO₂ photocatalysts

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Szeged

2014

1. Introduction and aims of the research

Advanced oxidation processes utilizing TiO₂ semiconductor photocatalysts can be used effectively for the degradation of numerous organic wastewater compounds. Photocatalytic disposal of wastewaters might be beneficial environmentally and economically if H₂ gas is produced at the same time. Furthermore this process can be attained with natural sunlight (4-5% UV and ~40% VIS component). Photons having the same or higher energy as TiO₂'s band-gap energy are needed for the excitation and photocatalytic transformation to take place. The wavelength of these photons are in the near UV-range, thus developing catalysts that can harvest visible light is substantial. Modifying the crystal structure of TiO₂ or depositing the surface with noble metal nanoparticles (e.g. Pt, Au, Ru or Rh) can lead to better utilization of the irradiated light, and lower overpotential of hydrogen evolution.

Photocatalytic H₂ generation using TiO₂ is controlled by a number of parameters: the type of noble metal deposited onto the catalyst's surface, the size of the nanoparticles, the size of TiO₂ particles and their crystal phase, the quality of organic compound being degraded, the temperature and the spectra of the irradiation. Many reaction parameters should be optimized for the process to be efficient and the combined effect of these conditions should be also investigated.

During this study I examined the applicability of *Aeroxide P25* and *Kronos vlp7000* TiO₂ photocatalyst for H₂ production. Au or Pt nanoparticles were deposited onto the catalyst's surface and the effect of noble metals to the photocatalytic processes was thoroughly investigated under UV irradiation. I was aimed to set out chemical reduction and photoreduction methods that can be used easily but efficiently to control the size and distribution of reduced noble metal nanoparticles. Comparing the results of the experiments I determined the optimal noble metal particle size and the variation of the evolved amount of H₂ gas was also studied.

A further aim was to find catalysts among numerous commercially available titanias that can be excited with photons above 400 nm and can be utilized for H₂ production with high efficiency. I was searching for the catalyst that provides the best H₂ generation rates when deposited with noble metal nanoparticles of its own and in combination with others. The influence of the organic support and the reaction temperature was also investigated.

2. Methods

2.1. Reactor used for the photocatalytic experiments

Photocatalytic activities of the catalysts were examined in 1 g/L suspensions poured into a reactor designed in our laboratory made of Pyrex glass. The temperature was set to a constant 25 °C that was established by distilled water when the light source was a set of UV lamps, and by 1 M NaNO₂ solution filtering <400 nm photons when visible light was applied. During the experiments N₂ gas was bubbled through the reactor at a flow rate of (mostly) 50 mL/min which was carrying the evolved gas to the *Hewlett Packard 5890 Type II* gas chromatograph. The GC was equipped with 5 Å molecular sieve column, 2 mL sampling valve and a thermal conductivity detector (TCD). The reactor was surrounded with ten lamps (15 W each, *LightTech*). UV lamps have an emission maximum at $\lambda_{\text{max}} = 365$ nm, while VIS lamps irradiated at maximum at $\lambda_{\text{max}} = 425$ nm wavelength.

2.2. Determination of total organic carbon (TOC) content

Samples were taken from the suspensions and injected directly into an *Analytik Jena multi N/C 3100* TOC equipment without centrifugation and filtering. The adsorbed and dissolved organic carbon content was measured simultaneously.

2.3. High performance liquid chromatography (HPLC)

A *Merck Hitachi L-4250 HPLC* equipped with a *UV-VIS* detector was used for the analytic measurement of undecomposed organic compounds. The filtered samples were injected manually to a *GROM Resin ZH* column, which was filled with 8 µm particles. The detection wavelength was 210 nm. Sulfuric acid of 19.3 mM was used as eluent.

2.4. Inductively coupled plasma spectrometry (ICP)

Concentration of Pt that may have been dissolved from the surface was determined by ICP-MS spectrometry (*Model 7700x, Agilent*) External calibration was used based on the signal from the ¹⁹⁵Pt isotope (*Certipur, Merck*).

2.5. Specific surface area of the catalysts (BET)

Specific surface area of the catalysts was determined according to the BET method, with nitrogen adsorption at 77 K, using a *Micromeritics Gemini Type 2375* device.

2.6. Spectrophotometry

The UV-VIS spectra of noble metal sols were measured in 1 cm quartz cells in an *Agilent 8453* diode array spectrophotometer, with *Millipore MilliQ* ultrapure water as blank reference.

2.7. Diffuse reflectance spectroscopy (DRS)

The UV-VIS reflection spectra ($\lambda = 300\text{-}800\text{ nm}$) of the photocatalyst powders were measured with a *JASCO-V650* spectrophotometer equipped with an integration sphere (*ILV-724*). The possible electron transitions were evaluated by plotting $dR/d\lambda$ vs. λ , where R is the reflectance and λ is the wavelength.

2.8. Transmission electron microscopy (TEM)

The average size and dispersion of the noble metal nanoparticles deposited on the TiO_2 surface was calculated from TEM images recorded with a 200 kV *Fei Tecnai G2 20 Xtwinn* instrument.

2.9. X-ray diffraction (XRD)

The average Au particle diameter was determined from the line broadening of the XRD peak of Au at 38.2° (2θ). XRD measurements were performed on a *Rigaku* diffractometer ($\text{CuK}\alpha = 0.15406\text{ nm}$, 30 kV, and 15 mA).

2.10. Energy-dispersive X-ray spectroscopy (EDX)

To determine the exact noble metal loading on the prepared TiO_2 samples SEM–EDX analysis was performed on a *Hitachi S-4700 Type II* cold field emission scanning electron microscope attached to a *Röntec QX2-EDS* spectrometer.

2.11. X-ray photoelectron spectroscopy (XPS)

To investigate the oxidation state of noble metal particles on TiO_2 surface XPS measurements were performed on a *Specs Phoibos 150 MCD* instrument, with monochromatized Al $\text{K}\alpha$ radiation (1486.69 eV) at 14 kV and 20 mA.

3. Thesis

I. The size of the forming gold nanoparticles can be gently controlled by the concentration of trisodium citrate when depositing Au nanoparticles onto TiO₂ surface with chemical reduction synthesis method (with the same gold content). For photocatalytic hydrogen production the presence of Au nanoparticles having ~6 nm average diameter is optimal [1].

I applied two types of synthesis methods for depositing Au nanoparticles onto TiO₂ surface: the reduction of gold took place *in situ* in the presence of TiO₂ (CRIS), or TiO₂ was impregnated with previously reduced gold sols (CRSIM). The average size of the formed Au nanoparticles was determined by the concentration of trisodium citrate stabilizing agent for both synthesis methods. Varying the concentration of stabilizer between 5.00×10^{-4} M and 0.63×10^{-4} M in five steps, I achieved the formation of particles of 2-8 nm in size (**Figure 1**). Increasing citrate concentration the average size of gold was decreased with diameters fluctuating in a small interval.

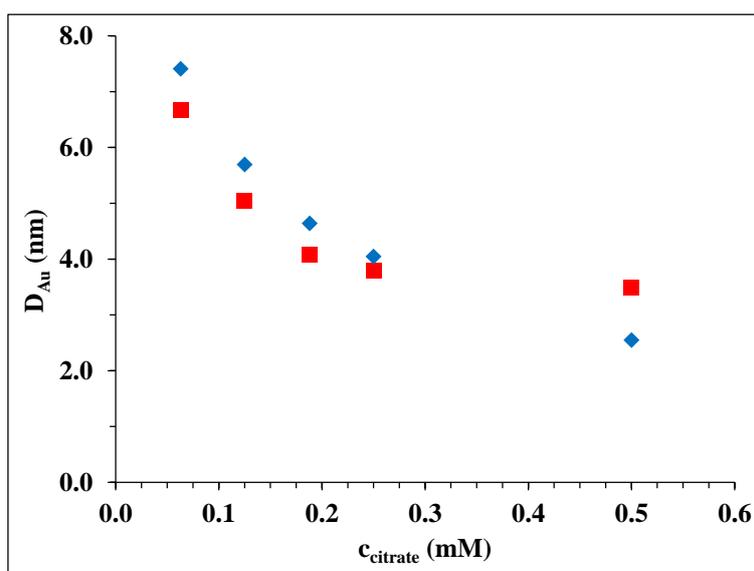


Figure 1.

Dependence of the average Au nanoparticle size on the citrate concentration during the synthesis (◆ Au-TiO₂(P25)-CRIS, ■ Au-TiO₂(P25)-CRSIM)

Gold content of Au-TiO₂ catalysts had a good correspondence with the theoretical 1 wt% according to EDX measurements, thus differences in the evolved amount of hydrogen can be originated in the size of gold nanoparticles presented on the surface of the catalysts. The largest amount of produced H₂ can be achieved when $D_{Au} \approx 6$ nm gold particles are deposited onto both *Aeroxide P25* and *Kronos vlp7000* in the presence of oxalic acid (initial concentration is 50 mM)

and UV irradiation (**Figure 2.**). With smaller Au nanoparticles the efficiency of charge separation is lowered due to the loss of metallic character, so the rate of redox reactions are also decreased [2-3]. Having too big Au nanoparticles on the surface can also lead to lower hydrogen generation rate due to the lowering the number of potentially active sites on the surface.

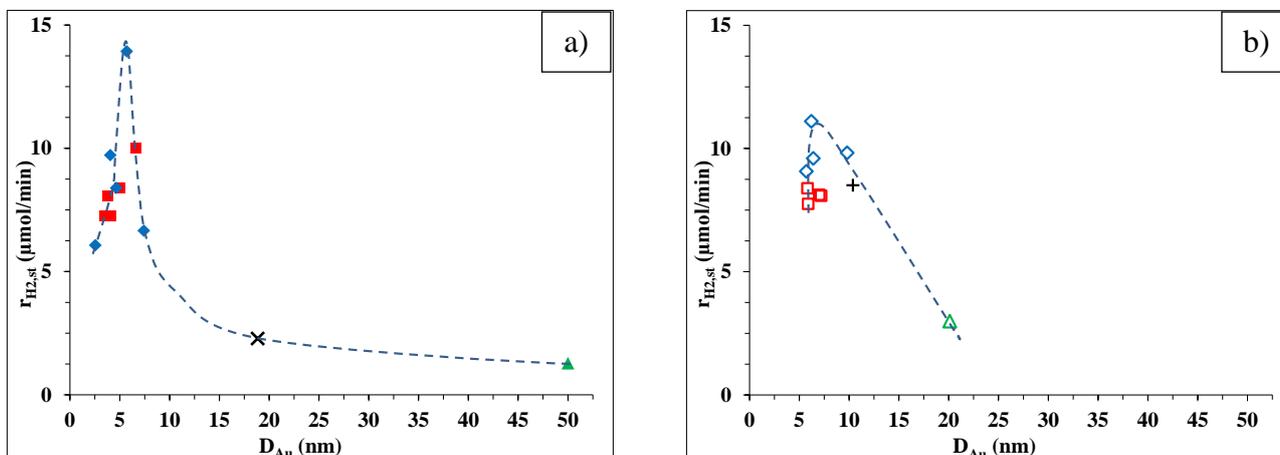


Figure 2.

Average H₂ evolution rates as a function of average Au particle size on different Au-modified TiO₂ photocatalysts

a) *Aerioxide P25* based samples (◆ CRIS; ■ CRSIM; × PRTC; ▲ PROA),
and b) *Kronos vlp7000* based samples (◇ CRIS; □ CRSIM; + PRTC; △ PROA)

II. Optimal Au distribution on the catalyst was achieved with *in situ* chemical reduction (CRIS) in the presence of TiO₂. Mixing gold sols with TiO₂ suspensions leads to less uniform presence of Au on the surface that can result in lower amounts of photogenerated hydrogen [1].

Analyzing the TEM images of gold modified *Aerioxide P25* the distribution of Au nanoparticles on TiO₂ was different according to the used synthesis method. Reduction of gold in the presence of TiO₂ resulted in nearly homogeneous distribution of Au on the catalyst's surface. However, noble metal nanoparticles were grouped on the surface and several TiO₂ particles did not have any gold on their surface when mixing the previously reduced gold sols with the titania suspensions. This phenomenon influenced the photocatalytic hydrogen production, too. Higher rates of evolving H₂ can be achieved with samples having optimally sized gold made by the CRIS method.

III. The large difference in the specific surface area does not affect the amount of evolving hydrogen when using gold modified *Aeroxide P25* or *Kronos vlp7000* TiO₂ photocatalysts. The rate of the photocatalytic reaction is limited by the adsorbed amount of oxalic acid and the size of Au nanoparticles [1].

There was only a small difference in the photocatalytic activities for H₂ generation using *Aeroxide P25* or *Kronos vlp7000* based samples deposited with gold of almost the same size. However, the specific surface area of the two types of titania differed significantly: 50.0 m²/g for *Aeroxide P25* and 296.5 m²/g for *Kronos vlp7000*. BET specific surface measurements proved that depositing TiO₂ with 1 wt% gold does not affect considerably this property (Au-TiO₂(P25): 48.7 m²/g; Au-TiO₂(VLP7000): 266.3 m²/g). Investigation of the oxalic acid adsorption revealed that *Kronos vlp7000* based samples bind almost the same amount of oxalic acid as *Aeroxide P25* based ones despite the six-fold difference in specific surface area. This may explain the slight distinction in the amount of produced H₂. Oxalic acid binds to the TiO₂ surface with dissociative adsorption [4] and it is oxidized mainly by the photogenerated holes [5], thus the adsorbed amount of oxalic acid might be rate determining factor for the redox reactions. *Kronos vlp7000* contains almost the same amount of active sites on the surface that can bind oxalic acid as *Aeroxide P25*.

IV. Average size of Pt particles deposited on TiO₂ photocatalysts has a remarkable impact on hydrogen production capabilities. Optimal catalytic activity can be achieved with *in situ* chemically reduced Pt modified *Aeroxide P25* with Pt of ~3.2 nm average diameter [6].

Aeroxide P25 based TiO₂ photocatalysts with 1 wt% Pt were synthesized. The size of the platinum particles was finely tuned by the concentration of trisodium citrate during the syntheses. The largest amount of H₂ was produced in the presence of oxalic acid and UV irradiation when ~3.2 nm Pt particles were deposited on the catalyst's surface. The presence of smaller or larger Pt particles are unfavorable and results in lower catalytic activities (**Figure 3**).

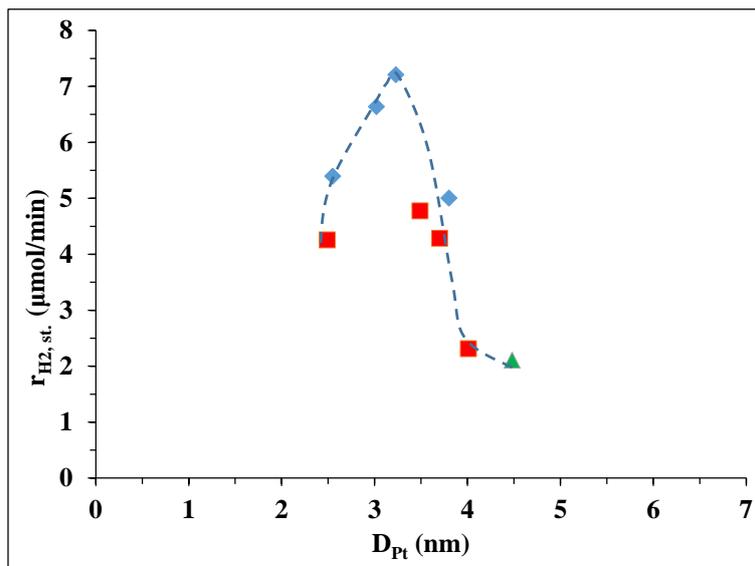


Figure 3.

Steady state ($t_{\text{irradiation}} > 50$ min) H_2 evolution rates in the presence of oxalic acid as a function of average Pt particle size Pt-TiO₂(P25) made by \blacklozenge CRIS; \blacksquare CRSIM and \blacktriangle PROA synthesis method

V. The significant difference among the H_2 evolution rates at the beginning and later stages of irradiation using Pt-TiO₂ in the presence of oxalic acid is caused by the absorption of hydrogen in platinum which leads to change of redox potentials. Dissolving of hydrogen in Pt is limited when higher carrier gas flow rate is applied, thus overpotential of hydrogen production can remain optimal for a longer period of time [6].

A significant decrease can be observed in H_2 production rates in the first 50 minutes of UV irradiation using Pt-TiO₂ photocatalysts. It was confirmed that the average particle diameter and size distribution of platinum does not change considerably during UV irradiation. Furthermore it was proven by ICP measurements that the detachment and dissolution of Pt from the TiO₂ surface is negligible. Raising the carrier gas flow rate (to 138 mL/min) causes an elongated maximum of H_2 production, while at low N₂ flow rate (25 mL/min) resulted in the complete disappearance of the initial maximum values in the investigated time frame. At later stages of the experiments ($t > 50$ min) the steady state H_2 evolution rates were identical. A possible explanation of the phenomenon can be that at the first period of irradiation the hydrogen generation is regulated by the varying potential of H-electrode established by the dissolution of hydrogen in Pt [7]. At higher carrier gas flow rates this process is limited, therefore the overpotential of hydrogen evolution can remain in an optimal range for a longer timeframe, while with lower N₂ flow the equilibrium redox potential can be achieved faster.

VI. 1 wt% noble metal deposited *Aldrich rutile* TiO₂ was the best-performing catalyst (among the six commercially available titanias that were tested) when visible light irradiation was used for the photocatalytic measurements. According to the type of deposited noble metals the photocatalytic enhancement was ordered as follows: Pt > Rh > Ru > Au. Increasing the reaction temperature to 80 °C resulted in much larger amounts of generated H₂ [8].

Only those 1 wt% Pt doped titanias can be utilized effectively for hydrogen production with visible light irradiation ($\lambda > 400$ nm) that contain rutile crystal phase along with or instead of anatase. TiO₂ particle size also has a significant role when >400 nm photons are used for excitation. According to the DRS spectras *Nanomaterials rutile* has much lower absorption in the visible range of lighth than *Aldrich rutile* (**Figure 4.**). This resulted in more than 2.5 fold difference in the H₂ production capabilities under the same reaction conditions using oxalic acid as sacrificial reagent.

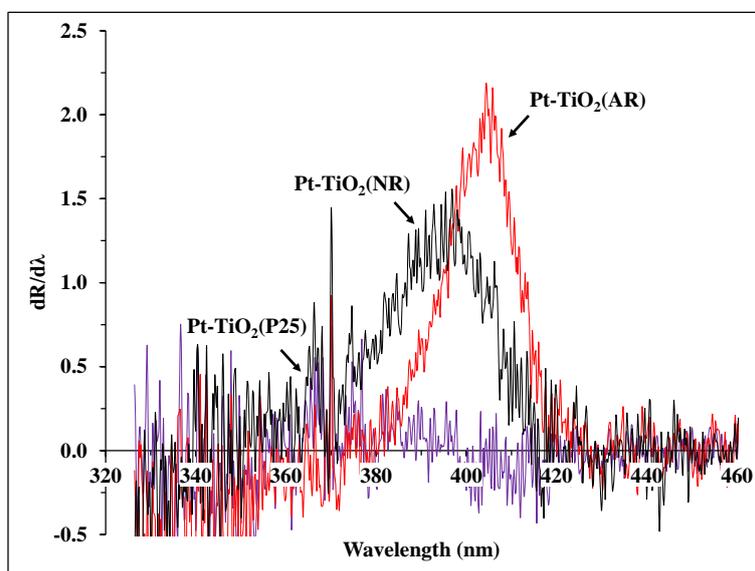


Figure 4.

Reflectance as a function of irradiation wavelength for Pt-modified rutile-containing TiO₂ photocatalysts (DRS measurements). Peaks represent the band gap energies of each catalyst

An explanation of this phenomenon is that *Nanomaterials rutile* has an average particle diameter of 30 nm while this value for *Aldrich rutile* is 315 nm. The excitation threshold and band-gap energy of semiconductors is shifted to shorter wavelengths when the particle size is too small due to phonon confinement [9].

Modifying the surface of *Aldrich rutile* TiO₂ with 1 wt% Au, Pt, Rh or Ru the largest amount of H₂ can be produced photocatalytically with the platinum modified catalyst, the activity is more than 3-fold higher than in the case of Au-TiO₂(AR) .

The amount of H₂ evolved on Pt-TiO₂(AR) in the presence of oxalic acid can be increased by applying higher reaction temperatures up to 80 °C. In practice, focusing the solar irradiation to a smaller surface might also lead to environmentally friendly heating of the suspension.

VII. The presence of organic aiding compounds with an acidic character resulted in the highest rates of hydrogen production when using Pt modified *Aldrich rutile* TiO₂ [8].

The quality of the organic aiding reactant presented in the reaction mixture seriously impacts the amount of evolved H₂ when Pt modified *Aldrich rutile* is irradiated with visible light. Using different simple organic compounds (oxalic acid, lactic acid, formic acid, methanol, ethanol, glucose and acetaldehyde) remarkable H₂ evolution can be expected in the solution of carboxylic acids (**Figure 5**).

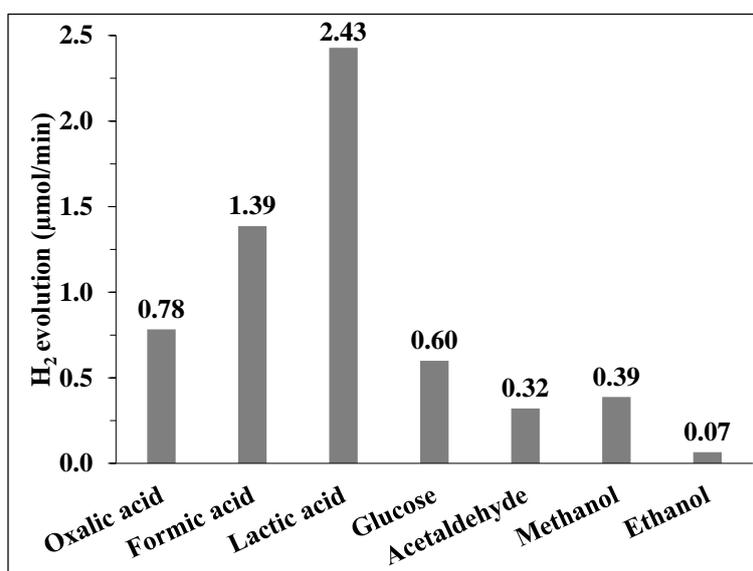


Figure 5.

H₂ evolution rates on Pt-TiO₂(AR) photocatalysts in the presence of various sacrificial reagents ($c_{\text{initial}} = 50$ mM) and VIS irradiation (averaged by the measured rates between 120-240 min)

Due to their acidic dissociation, they bind stronger to the –OH groups on the surface of TiO₂ that leads to a higher chance to react with photoinduced holes and radicals adsorbed on the catalyst's surface.

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SCIENTIFIC ACTIVITY

Published / in press papers:	4 (Cumulative impact factor: 9,861)
Citations:	26 (Independent citations: 21)
Conferences:	
As speaker:	8
As co-author:	5
Poster presentations:	7
Book chapters:	6

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