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Using hydrogen and oxygen in water directly for hydrogenation reactions and glucose oxidation by photocatalysis

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Direct utilization of the abundant hydrogen and oxygen in water for organic reactions is very attractive and challenging in chemistry. Herein, we report the first work on the utilization of the hydrogen of water for hydrogenation of various organic compounds to form valuable chemicals and the oxygen for the oxidation of glucose simultaneously by photocatalysis. It was discovered that various unsaturated compounds could be efficiently hydrogenated with high conversion and selectivity by the hydrogen from water splitting and glucose reforming over Pd/TiO₂ under UV irradiation (350 nm). At the same time, glucose was oxidated by the hydroxyl radicals from water splitting and the holes caused by UV irradiation to form biomass-derived chemicals, such as arabinose, erythrose, formic acid, and hydroxyacetic acid. Thus, the hydrogen and oxygen were used ideally. This work presents a new and sustainable strategy for hydrogenations and biomass conversion by using the hydrogen and oxygen in water.

utilization is a long term task.

Introduction

Efficient utilization of hydrogen and oxygen in water is one of the most attractive but challenging technologies for the sustainable development of our society. Photocatalytic water splitting offers a fascinating means for the production of H₂ and O_2 from water.^{1,2} Over the past decades, considerable efforts have been devoted to this area, and great progress has been made.³ However, many challenging problems are still unsolved, such as low energy efficiency, environment-sensitive reaction systems, separation of H₂ and O₂, and efficient storage, transportation and combustion of H₂, etc. Therefore, it is highly attractive to develop effective and novel routes for the utilization of hydrogen and oxygen from water splitting. Using the abundant hydrogen and oxygen in water directly for hydrogenation and oxidation is a desirable strategy.

Lignocellulosic biomass is an abundant and renewable resource.⁴ Glucose can be obtained from depolymerization of cellulose, which is a main component of lignocellulosic biomass.⁵ Conversion of glucose into various value-added chemicals and fuels has received much attention.⁶ Furthermore, H₂ can also be generated from glucose by various routes, including photocatalytic process,⁷ catalytic reforming,⁸ and enzymatic procedures.⁹ Up to now, attention has been mainly focused on transformation of glucose into

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chemicals or H₂. Exploration of new processes for glucose

various chemicals. Current hydrogenation processes usually

suffer from harsh reaction conditions and lower selectivity.

Furthermore, reduction processes that use stoichiometric

reductants generally form large amounts of environment-

milder reaction conditions. Triethanolamine,^{16,17} isopropanol,¹⁸

Use of cheap and abundant resources in

hydrogenation of organic compounds and transformation of

simultaneously by photocatalysis. In this approach, the

organic compounds, and at the same time the oxygen in water

donors, which makes the process less sustainable.

which the main products are provided.

Hydrogenation is an important approach for producing

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Scheme 1. Photocatalytic water splitting for hydrogenation of various organic compounds and conversion of glucose to valuable chemicals.

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Results and discussion

Selective hydrogenation of nitrobenzenes containing other reducible groups provides a convenient route to produce functionalized anilines, which are high-value intermediates for various fine chemicals. Thus, photocatalytic reduction of functionalized nitrobenzenes was selected to study the reaction in aqueous solution of glucose by UV-light (350 nm). Pd/TiO₂, which was effective for photocatalytic hydrogenation of the nitro group in the presence of various alcohols, ^{20,21} was utilized as the catalyst. Transmission electron microscope (TEM) examinations indicated that the difference between the size of the Pd nanoparticles (PdNPs) in the Pd/TiO₂ catalysts with Pd loadings of 1 wt%, 2 wt%, and 3 wt% was not considerable (Fig. S1). Fig.1 (a-c) shows the influence of Pd contents in the Pd/TiO₂ catalysts on photocatalytic reduction of 4-nitroacetophenone to 4-aminoacetophenone. It was found that the catalyst with 2 wt% Pd had better catalytic performance than those with 1 wt% and 3 wt% Pd. The main reason is that the loading amount of Pd affected the reaction in opposite ways. In the catalytic cycle, Pd nanoparticles served as electron sinks and provided active sites for forming H-PdNPs, which is the active species for the reduction of the nitro group. On the other hand, excessive loading of Pd nanoparticles could reduce the photocatalytic activity because Pd could also act as charge carrier recombination centers,²² and shielded the incident light absorption by the semiconductor.²³ The competition of the above factors resulted in the best performance of the catalyst with 2 wt% Pd content. The 4-nitroacetophenone could be completely converted in 12 hours (Fig. 1a) with a 4-aminoacetophenone selectivity of 98% (Fig. 1b).

Fig. 1 (d-f) illustrates the influence of the molar ratio of glucose to substrate on photocatalytic hydrogenation of 4nitroacetophenone with different reaction times. It can be seen that the conversion of 4-nitroacetophenone (Fig. 1d), the selectivity (Fig. 1e) and yield (Fig. 1f) of 4-aminoacetophenone increased with the increasing molar ratio of glucose to substrate. In the reaction process, glucose performed as the scavenger of the photoexcited holes to inhibit the recombination of the electron-hole pairs on TiO_2 . More importantly, photocatalytic reforming of glucose with the aid of hydroxyl radical from water splitting could release protons, which could be reduced by photoinduced electrons to form the active H-PdNPs species, which will be discussed in detail in the subsequent sections. Therefore, increasing the molar ratio of glucose to substrate facilitated the elimination of the holes and promoted the formation of H-PdNPs species, which was beneficial to the photocatalytic reduction. Our control experiment showed that the hydrogenation reaction could be prohibited by the addition of 2,2',6,6'-tetramethylpiperidine N-oxyl (TEMPO, 100 mg), which could remove hydrogen atom from the metal surface,²⁴ indicating the formation of H-PdNPs. In addition, when the molar ratio of glucose to substrate was increased to 100/1, the reaction time for the complete conversion of 4-nitroacetophenone was reduced to 4 hours with a 4-aminoacetophenone selectivity of 98%, which further

proved that high molar ratio of glucose to substrate was beneficial for the reaction. DOI: 10.1039/C5SC03178H



Fig. 1. Effect of the Pd contents in the Pd/TiO₂ catalysts (a-c) and the molar ratio of glucose to substrate (d-f) on the photocatalytic reduction of 4-nitroacetophenone to 4-aminoacetophenone. Reaction conditions: 4-nitroacetophenone, 0.1 mmol; water, 1 mL; 0.025 g Pd/TiO₂ with various Pd contents; UV-light (350 nm, 4 mW/cm²); illuminated area, 2 cm²; temperature, 25 °C. The molar ratio of glucose to substrate was 5 in a-c; Pd/TiO₂ with 2 wt% Pd was used in d-f.

Water splitting accompanied with glucose reforming continuously proceeded over Pd/TiO₂ under UV-light illumination, releasing protons and electrons for photocatalytic hydrogenation. As shown in Fig. 2 and Table 1, 0.13 mmol of glucose were consumed for total conversion of 0.1 mmol of 4nitroacetophenone to 4-aminoacetophenone, as was known from HPLC analysis with a refractive detector (Shimadzu RID-10A). Simultaneously, a series of biomass-derived chemicals, including arabinose, erythrose, formic acid, and hydroxyacetic acid were formed from the consecutive photoxidation o glucose by the oxygen in the form of hydroxyl radicals from water and the holes on TiO₂ caused by UV irradiation. In order to confirm that the hydrogen in water took part in the reaction, we carried out the control experiment using D_2O to replace H₂O with a reaction time of 12 hours and other conditions were the same as that shown in Fig. 1b. The -NO₂ was reduced to -NH₂, -NDH, and -ND₂ with the -NH₂:-NDH:-ND₂ ratio of 0.60:0.32:0.08, indicating that 24% hydrogen was from water in the reaction. This indicates that the hydrogen for the reduction was derived from both water splitting and glucose reforming. Our experiment showed that the reaction did not occur without glucose, indicating that to use the hydrogen of water for the hydrogenation reaction, the oxygen of water must be consumed in oxidation of glucose simultaneously,

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which will be discussed in the following sections. In addition, hydrogenation did not occur in dark or when the wavelength of incident light was >420 nm. The reason was that the electron-hole separation resulting from the wide band gap of TiO₂ (3.2 eV) could not be induced, which was the first key step for the subsequent photoredox reactions. Furthermore, only very little amount (about 2%) of 4-aminoacetophenone was produced upon simulated sunlight because UV light energy only contributed to little proportion (about 4%) of solar energy. These results suggested that UV-light was the driving force of the reaction when Pd/TiO₂ was used as the catalyst.



Fig. 2. The dependence of amounts of the products from glucose reforming in water on time at the reaction conditions of Fig. 1b.

Experiments were also conducted to examine the reusability of Pd/TiO₂. In each cycle, Pd/TiO₂ was recovered by centrifugation and washed by ethanol. After dried at 60 °C under vacuum for 12 hours, the catalyst was reused for the next run. The conversion of 4-nitroacetophenone and the yield and selectivity of 4-aminoacetophenone are shown in Fig. 3. It was found that there was no considerable decrease in the conversion, selectivity, and yield after four cycles, indicating that Pd/TiO₂ was stable in the reaction. Meanwhile, Pd/TiO₂ recovered after reused four times was characterized by TEM, and the result indicated that the size of the Pd particles was not changed obviously (Fig. S4).



Fig. 3. Reusability of Pd/TiO₂ for photocatalytic reduction of 4-nitroacetophenone in glucose aqueous solution. Reaction conditons: 4-nitroacetophenone, 0.1 mmol; glucose, 0.5 mmol; water, 1 mL; temperature, 25 °C; reaction time, 12 h; 0.025 g Pd/TiO₂ with 2wt% Pd; UV-light irradiation (350 nm, 4 mW/cm²); illuminated area, 2 cm².

Photocatalytic reduction of nitrobenzenes with warious substituted reducible groups was performed, and the cestilits are presented in Table 1. It was found that all the nitrobenzenes examined could be converted to the corresponding anilines in the water-glucose system. The activity order of para-, meta- and ortho-substituted nitroacetophenone (Entries 1-3, Table 1) was para- > meta->ortho- caused mainly by the steric hindrance. Meanwhile, 4iodo-nitrobenzene (Entry 8, Table 1) and 4-chloronitrobenzene (Entry 9, Table 1) showed lower selectivity because of the dehalogenation reaction occurred in the reaction process. In addition, nitroethane (Entry 11, Table 1) could be reduced to ethylamine with a selectivity of 97% with a prolonged reaction time (48 h), indicating that our reaction system could also be used to reduce nitroalkanes.

 $\mbox{Table 1.}$ Photocatalytic selective reduction of various nitro compounds in water-glucose system. $\mbox{}^{\rm a}$

Entry	Substrate	Time	Nitroarenes	Anilines	Glu. cons.	
Lindiy	Substrate	(h)	Con.(%) ^b	Sel.(%) ^c	(mmol) ^d	
1	H ₃ COC-O-NO ₂	12	>99	99	0.11	+
2	COCH3	12	92	89	0.13	
3	H ₃ COC	12	98	96	0.12	Ç
4	OHC	11	>99	98	0.13	5
5		10	>99	99	0.11	
6	H2C=HC-VO2	16	98	93	0.12	9
7	HC=C-	18	>99	96	0.12	Ì
8		16	97	88	0.13	
9		16	95	90	0.13	C
10	C2H500C-	20	98	96	0.12	Ù
11	∕_ _{NO₂}	48	95	97	0.20	

^aReaction conditions: substrates, 0.1 mmol; glucose, 0.5 mmol; water, 1 mL; temperature, 25°C; 0.025 g Pd/TiO₂ with2 wt% Pd; UV-light (350 nm, 4 mW/cm²); illuminated area, 2 cm². ^bCon.=Conversion. ^cSel.=Selectivity. ^dGlu. cons.= Glucose consumption.

From Table 1, it was found that the substituted nitrobenzenes with other reducible groups could be reduced to the corresponding anilines with good to excellent (88-99%) selectivity by economic consumption of glucose. The high selectivity can be attributed mainly to two reasons. The first is that the nitro group prefers to be absorbed on the catalysts in comparison with other reducible groups,¹⁰ which was beneficial to selective reduction of the nitro group. A kinetic

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experiment was conducted to prove this. When the mixture of nitrobenzene (0.1 mmol) and benzyaldehyde (0.1 mmol) was used as the reactants, it was found that nitrobenzene was preferentially hydrogenated in the process, while the reduction of benzyaldehyde was inhibited completely in the presence of nitrobenzene (Fig. S2). Secondly, the nitro group has stronger electron-withdrawing ability than other substituted groups, exhibiting better electrophilicity,¹⁶ which is favorable to the reduction of the nitro group by the active H-PdNPs species compared with other reducible groups. We designed and carried out the control experiments to find evidence to support this argument. In the experiment, the photocatalytic hydrogenation of nitrobenzene (0.1 mmol), benzyaldehyde (0.1 mmol) and acetophenone (0.1 mmol) were performed separately in 2 mol/L glucose aqueous solution with a reaction time of 6 hours under UV-light (350nm), and the results are illustrated in Fig. S3. It was found that the nitro group had a higher reaction rate than the aldehyde and carbonyl groups. This suggests that the nitro group had a high intrinsic catalytic activity than other reducible groups.

Generally, there are two possible pathways for hydrogenating nitrobenzenes to anilines (Fig. 4). As shown by HPLC analysis, there were no azoxybenzene and azobenzene produced during the entire catalytic process. In contrast, nitrosobenzene and hydroxyamine were formed. These results indicated that the photocatalytic reduction of nitrobenzenes to the corresponding anilines was through the pathway A in our reaction system (Fig. 4). On the basis of the experimental results and some related reports, ^{10, 25, 26} we propose a possible mechanism about photocatalytic selective reduction of nitrobenzenes to anilines in water-glucose system over Pd/TiO₂, which is shown in Fig. 5 and Fig. 6. Firstly, electronhole separation on TiO₂ occured under UV-light irradiation (Fig. 5).Then, water splitting accompanied with photocatalytic reforming of glucose was induced by the photogenerated holes to release protons and hydroxyl radicals (Fig. 5). The hydroxyl radical involved photocatalytic reforming of glucose. A series of biomass-derived chemicals were formed through C1-C2 cleavage from the continuous photoxidation of glucose by the hydroxyl radicals from water splitting and the holes on TiO₂ caused by UV irradiation (Fig. 6). Subsequently, the photoinduced electrons could readily reduce H⁺ from water splitting and glucose reforming on PdNPs to form the active H-PdNPs species (Fig. 5). Finally, the nitro group of nitrobenzenes is preferably absorbed on the catalyst, and predominantly reduced to the functionalized anilines by the H-PdNPs species through the Pathway A shown in Fig. 4.



Fig. 4. Possible pathways of photocatalytic selective reduction of functionalized nitroarenes to anilines.



Fig. 5. Proposed mechanism of photocatalytic water splitting and glucose reforming for selective reduction of nitrobenzenes to anilines catalyzed by Pd/TiO₂.



Fig. 6. Proposed routes of photocatalytic water splitting and glucose reforming over Pd/TiO $_{2}$.

Inspired by the results obtained from the selective photocatalytic reduction of nitrobenzenes, we explored hydrogenation of other kinds of unsaturated organic compounds using the same method, and the results were illustrated in Table 2. It was found that the hydrogen from water splitting and glucose reforming could be used for efficiently hydrogenating benzyaldehdye and hydroazobenzene to the corresponding saturated products with high yields. The products from glucose were also mainly arabinose, erythrose, formic acid, and hydroxyacetic acid.

Control reactions conducted in D_2O showed that the contents of deuterohydrogen in the products were 39% and 30%, respectively. This result further proved that the hydrogen for the reductions originated from both water and glucose.

Table	2.Photocatalytic	water	splitting	and	glucose	reforming	for	various
hydrog	enations. ^a							

Entry	Reactant	Product	t (h)	Con. (%) ^b	Yield (%)	Hydrogen from water
1	Срено	∕⊂≻−сн₂он	24	96	89	39%
2			10	100	90	30%

^aReaction conditions: substrates, 0.1 mmol; glucose, 0.5 mmol; water, 1 mL; temperature, 25 $^{\circ}$ C; 0.025 g Pd/TiO₂ with 2 wt% Pd; UV-light (350 nm, 4 mW/cm²); illuminated area, 2 cm². ^bCon.=conversion.

Conclusions

In summary, direct utilization of the hydrogen and oxygen of water for organic reactions was realized by photocatalysis. It was discovered that nitrobenzenes with different reducible groups could be efficiently reduced to the corresponding anilines with high selectivity by the hydrogen from water splitting and glucose reforming in the presence of Pd/TiO₂ under UV irradiation (350 nm). Control experiments in D₂O indicated that the hydrogen for the hydrogenation of nitrobenzenes from water was about 24%. Meanwhile, glucose was oxidated to various biomass-derived chemicals through C1-C2 bond cleavage, such as arabinose, erythrose, glycolic acid, and formic acid, by the hydroxyl radicals from water splitting and the holes on TiO_2 caused by UV irradiation. Further study indicated that consumption of the oxygen of water in the oxidation of glucose is necessary for the hydrogenation reactions.

Experimental

Preparation of Pd/TiO₂ with different amounts of Pd

The supported catalysts were synthesized by photoreduction method, which was similar to that reported in the literature.²⁷ In a typical experiment, 5 mL of water, 0.090 g of glucose, 0.025 g of TiO₂, and desired amounts of PdCl₂ were added into a stainless-steel reactor of 10 mL with a top-irradiation quartz window. The Pd/TiO₂ was obtained after photocatalytic reduction of the Pd(II) by UV irradiation (λ = 350 nm, light intensity 4 mW/cm²) for 1 h.

Photocatalytic Reactions

The photocatalytic reaction was carried out in a cylindrical stainless-steel reactor of 10 mL. There was a quartz window at the top of the reactor for the light irradiation. In an experiment, desired amounts of water, catalyst, glucose, and substrate were added into the reactor. After degassing, the reactor was irradiated by a Xenon lamp equipped with an optical filter for a desired reaction time at 25 $^{\circ}$ C, and the illuminated area was 2 cm². The photoreduction products were

analyzed by HPLC with Shimadzu LC-15C pump, Shimadzu UM-Vis SPD-15C detector, and a Hypersil ODS2 roluma at 35.7Cc Methanol/water solution (60/40 V/V) was used as the mobile phase at a flow rate of 1.0 mL/min. The chemicals in the reaction mixture were identified by GC-MS (QP-2010) as well as by comparing retention times to the respective standards in HPLC traces.

Reusability of Pd/TiO₂

In the experiments to test the reusability of Pd/TiO_2 , the catalyst was recovered by centrifugation, and washed with ethanol. After drying under vacuum at $60^{\circ}C$ for 12 h, the catalyst was reused for the next run.

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