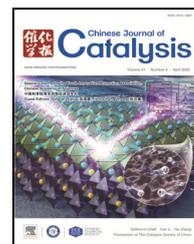


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Communication (Special Column for the Youth Innovation Promotion Association, Chinese Academy of Sciences)

Unexpectedly selective hydrogenation of phenylacetylene to styrene on titania supported platinum photocatalyst under 385 nm monochromatic light irradiation



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ABSTRACT

Conversion of alkynes to alkenes by photocatalysis has inspired extensive interest, but it is still challenging to obtain both high conversion and selectivity. Here we first demonstrate the photocatalytic conversion of phenylacetylene (PLE) to styrene (STE) with both high conversion and selectivity by using the titania (TiO₂) supported platinum (Pt) as photocatalyst under 385 nm monochromatic light irradiation. It is demonstrated that the conversion rate of PLE is strongly dependent on the content of Pt cocatalyst loaded on the surface of TiO₂. Based on our optimization, the conversion of PLE and the selectivity towards STE on the 1 wt% Pt/TiO₂ photocatalyst can unexpectedly reach as high as 92.4% and 91.3%, respectively. The highly selective photocatalytic hydrogenation can well be extended to the conversion of other typical alkynes to alkenes, demonstrating the generality of selective hydrogenation of C≡C over the Pt/TiO₂ photocatalyst.

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Alkenes are the key building blocks in various natural products, pharmaceuticals and agrochemicals, which have been widely investigated and synthesized by selective hydrogenation of alkynes [1,2]. Meanwhile, one of the main challenges is to suppress the over-hydrogenation of alkynes to alkanes. To achieve high selectivity of the alkyne semi-hydrogenation, the conventional thermodynamic catalytic reaction has developed popular Lindlar's catalyst, palladium (Pd) supported on calcium carbonate, by employing flammable H₂ as the reducing agent to add two hydrogen atoms to the C≡C of alkynes [3]. In this case,

the introduction of high pressure of H₂ may cause the danger of explosion [4–7]. Therefore, an environmentally friendly catalytic system for the selective hydrogenation of alkynes to alkenes working free from H₂ is highly desirable.

Photocatalysis is a promising approach to replace the conventional thermodynamic catalysis by utilizing the active photogenerated electrons and holes to participate organic reactions [8–10]. In consideration that the potential explosion danger of high pressure H₂ can be avoided through a mild hydrogenation reaction, where organic molecules are employed as

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hydrogenation source, such as isopropanol, triethanolamine, and methanol [11–14]. Due to the environmentally friendly application of TiO_2 photocatalysis, various photocatalytic reactions over TiO_2 photocatalyst have been reported [15–19], especially for the photocatalytic reduction of organic compounds by using the photogenerated electrons. For example, it was reported that nitrobenzenes having reducible group such as vinyl group are selectively reduced over bare TiO_2 photocatalyst to the corresponding aminobenzenes [14]. Kominami et al. [20] reported that the copper loading on the surface of TiO_2 photocatalyst is effective to improve the selective hydrogenation of 4-octyne with 2-propanol as the hydrogenation source. Xiong and co-workers [21] reported a selective light-driven 2-methyl-3-butyn-2-ol semi-hydrogenation reaction by using PdPt alloy as cocatalyst loaded on TiO_2 photocatalyst. Although various selective hydrogenation reactions have been reported with TiO_2 photocatalyst, an effective photocatalytic selective hydrogenation of PLE on TiO_2 has not been reported yet. Herein, we report the mild photocatalytic conversion of PLE to STE with both high conversion and selectivity by using the TiO_2 supported Pt as photocatalyst and methanol as hydrogenation source under 385 nm monochromatic light irradiation. The highly selective hydrogenation can be well extended to the conversion of other typical alkynes to alkenes, demonstrating its generality.

The cocatalyst of Pt nanoparticles was loaded on the surface of TiO_2 by the photodeposition method, and the corresponding sample was denoted as Pt/ TiO_2 (1 wt% Pt). Fig. 1(a) and Fig.

1(b) show the typical transmission electron microscopy (TEM) images of the pristine TiO_2 and Pt/ TiO_2 . It clearly shows that Pt nanoparticles are uniformly deposited on the surface of TiO_2 by photodeposition method, and the size of Pt nanoparticles is about 5 nm. X-ray diffraction patterns (XRD) in Fig. 1(c) indicate a mix phase of anatase (JSPDS #21-1272) and rutile (JSPDS #21-1276) for TiO_2 . While for the Pt/ TiO_2 sample, no obvious Pt peaks are observed due to the few amount of cocatalyst on the semiconductor. It is general to know that the TiO_2 is a widely investigated UV-responsive photocatalyst with absorption edge of ca. 400 nm [22–25]. The UV-vis absorption spectra in Fig. 1(d) shows the absorption edge of TiO_2 is consistent with previous work [26]. After loading Pt cocatalyst, the absorption intensity between 400 to 800 nm increases as compared to TiO_2 , which can be attributed to the loading of Pt cocatalyst.

The photocatalytic hydrogenation of PLE was performed in the methanol suspension of 1.0 wt% M/ TiO_2 with 385 nm monochromatic light (“M” stands Pt, Pd, Au, Rh and Ag). As shown in Fig. S1, the Pt/ TiO_2 catalyst shows the best hydrogenation performance of PLE and high selectivity towards STE. Fig. 2(a) shows the time-dependent curves of photocatalytic hydrogenation of PLE on the Pt/ TiO_2 photocatalyst, in which the photocatalytic conversion rate of PLE is almost linearly increased as a function of irradiation time. And the selectivity towards STE is maintained for 91.3% at the experimental region. Conversion rate of PLE reaches 100% after 8 h irradiation, which is consistent with the decreased amounts of PLE in

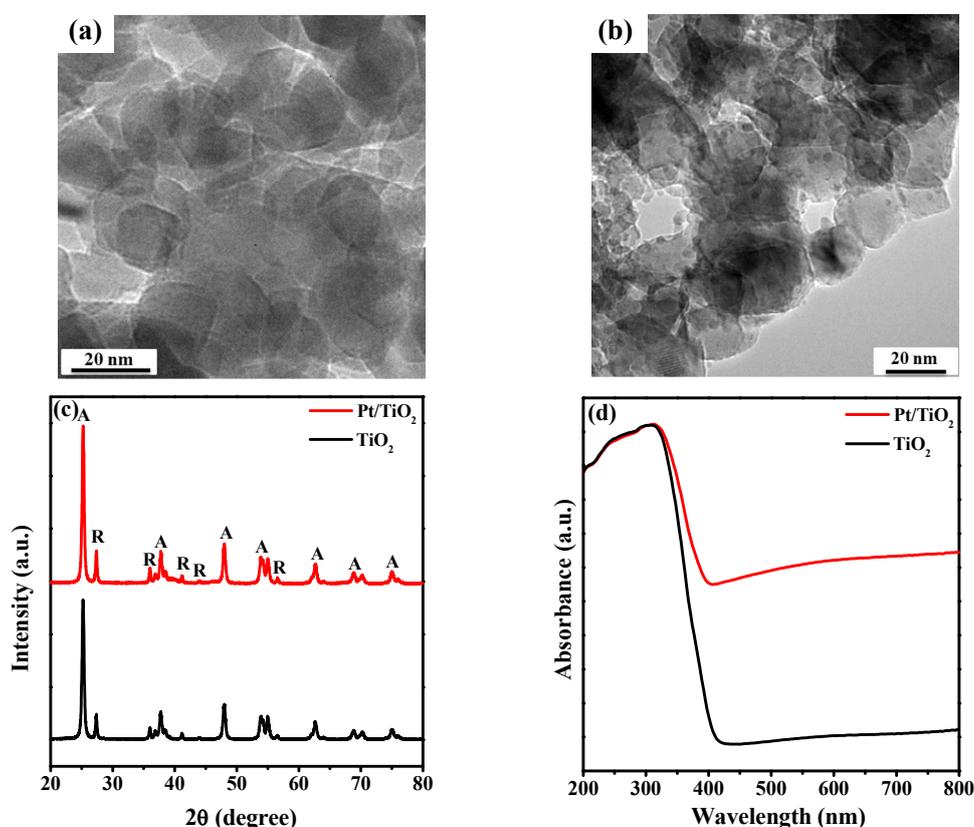


Fig. 1. TEM images of TiO_2 (a) and Pt/ TiO_2 (b); (c) XRD patterns of TiO_2 and Pt/ TiO_2 , A: anatase; R: rutile; (d) UV-visible absorption spectra of TiO_2 and Pt/ TiO_2 .

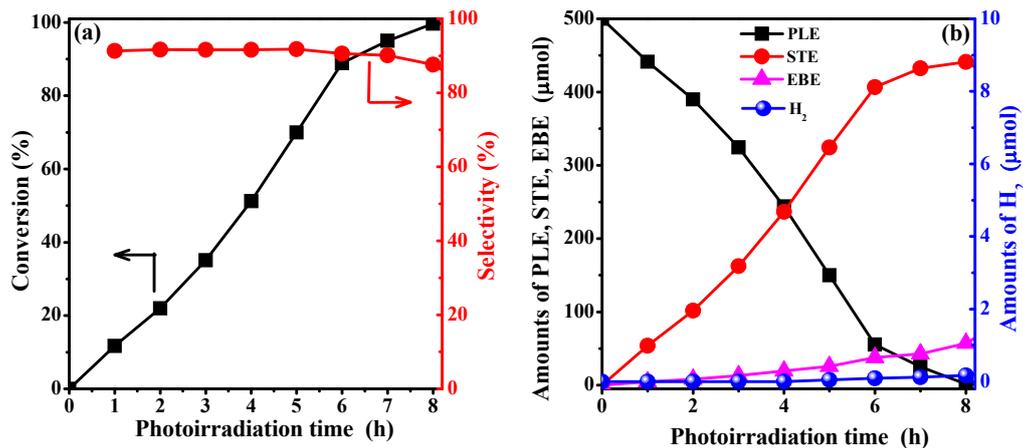


Fig. 2. Time courses of the conversion of PLE hydrogenation and the selectivity towards STE (a), and the amounts of PLE, STE, EBE and the amounts of H₂ (b) produced in the methanol suspension of Pt/TiO₂. Reaction conditions: PLE 0.5 mmol, Pt/TiO₂ 50 mg, methanol 25 mL, LED irradiation ($\lambda = 385$ nm) 5 mW·cm⁻².

Fig. 2(b). PLE is almost completely consumed after 8 h. The selectivity of STE in the products always remains at 91.3% before 6 h. But it decreases after 6 h photoirradiation, indicating the intermediate product of STE is hydrogenated further to EBE after a prolonged reaction time. As shown in Fig. 2(b), the amount of STE increases slowly after 6 h irradiation, while the amount of EBE increases obviously. It is clear to see that the intermediate product of STE is hydrogenated further to EBE after a prolonged irradiation time. The TiO₂ sample without Pt has no hydrogenation activity towards PLE (as shown in entry 4, Table S1). It is inferred that the cocatalyst is essential for the photocatalytic hydrogenation of the PLE substrate. It should be pointed out that the byproduct of H₂ is barely observed in Fig. 2(b).

The effect of the Pt loading content was also investigated in Fig. 3(a). The conversion rate of PLE increases with adding the Pt amount up to 1.0 wt%, which is due to the increase of active sites for hydrogenation reaction. With the amount of Pt in-

creases to 1.5 wt%, the conversion rate decreases because of the light-shading effect of the excessive cocatalyst. To illustrate the process of hydrogen evolution, the chemical states of deposited Pt are further examined by X-ray photoelectron spectroscopy (XPS). According to the binding energies shown in Fig. 3(b), the deposited Pt by photodeposition method exists as both metallic Pt and PtO_x. It is reported PtO_x is less reactive for H₂ evolution than metallic Pt [26–28]. The Pt/TiO₂ sample prepared by photodeposition method in Fig. 3(b) shows more PtO_x. According to the peaks area of metallic Pt and PtO_x, the content of PtO_x is calculated to be 59% approximately, which is disadvantageous for H₂ evolution. The byproduct is only EBE.

For comparison, the comparative experiment is performed over Pt/TiO₂ using H₂ as the hydrogenation source (1 atm) at room temperature free of light irradiation. The reaction time (6 h) is similar to that of photocatalytic hydrogenation. Interestingly, the result in Fig. 4 shows that EBE is detected as the only product. The conversion rate of PLE and the selectivity towards

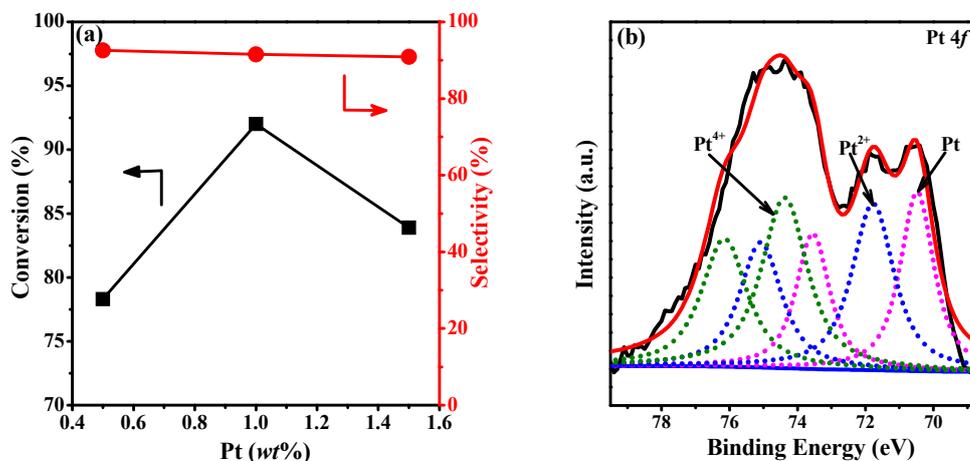


Fig. 3. (a) Effect of amounts of Pt loading on TiO₂ on the photocatalytic hydrogenation of PLE (0.5 mmol) in methanol suspension for 6 h under 385 nm monochromatic light; (b) XPS spectra of the Pt 4f of Pt/TiO₂.

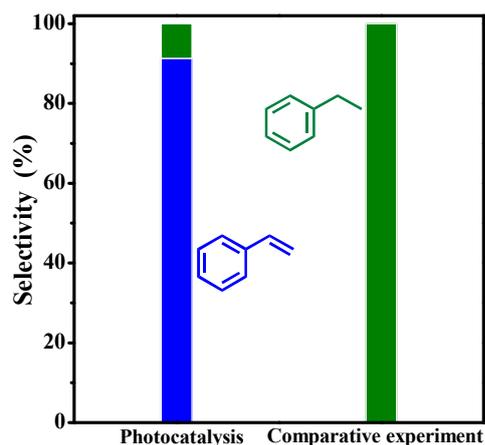


Fig. 4. Comparison of the selectivity towards STE of photocatalytic hydrogenation of PLE and comparative experiment.

EBE are both 100%, which is in sharp contrast with the photocatalytic hydrogenation result. The comparison clearly shows that the photocatalytic hydrogenation of PLE to STE has high selectivity of 91.3%, demonstrating the unique superiority of the photocatalytic hydrogenation method. What's more, the photocatalytic selective hydrogenation of alkynes to alkenes is working at room temperature free of direct H₂ source. A series of blank experiments related to the light irradiation and usage of photocatalyst were also carried out with results given in Table S1, based on which both Pt/TiO₂ and UV light are demonstrated to be indispensable for the photocatalytic hydrogenation of PLE to STE.

To examine the generality of selective hydrogenation of alkynes over the Pt/TiO₂ photocatalyst, other aromatic and ali-

Table 1

Photocatalytic semi-hydrogenation of alkynes to the corresponding alkenes in the methanol suspension of Pt/TiO₂ at room temperature ^a.

Entry	Alkyne	Time (h)	Con. ^b (%)	Alkene ^c	Select. ^b (%)
1		6	92.4		91.3
2		6	76.8		93.2
3		6	26.2		89.4
4		6	44		100
5		6	76.6		87.9

^a Reaction conditions: Pt/TiO₂ (50 mg), substrate (0.5 mmol), methanol (25 mL), 385 nm LED monochromatic light, room temperature, Ar atmosphere.

^b The conversion of alkynes and the selectivity of corresponding alkenes were determined by GC equipped with a DB-5 column using mesitylene as an internal standard.

^c The produced alkenes were determined by gas chromatography-mass spectrometry (Shimadzu GCMS-QP2010 SE) with a RXI-5MS column (30 m, 0.25 mm i.d., stationary phase thickness of 0.25 μm).

phatic alkynes were investigated. As seen in Table 1, the Pt/TiO₂ photocatalyst is effective for selective hydrogenation of aliphatic and aromatic alkynes to the corresponding *cis*-alkenes (Table 1, entries 1–5), well demonstrating its generality. The conversion of alkynes decreases when the functional groups attach to benzene ring or triple bond (entries 2 and 3) compared with PLE, which is possibly caused by the decreased adsorption of substrate on the surface of photocatalyst due to steric hindrance. Alkynes having chloro group is also converted into the corresponding alkenes, and the chloro group is preserved (entry 5). The photocatalytic hydrogenation activity of the aliphatic alkyne having longer carbochain is a little lower (entries 4 and 5).

Fig. 5 schematically illustrates the photocatalytic hydrogenation of PLE and comparative experiment over the Pt/TiO₂ catalyst. As shown in Fig. 5(a), the main product of photocatalytic hydrogenation of PLE is STE. While the product of the comparative experiment is only EBE in Fig. 5(b). The detailed reaction mechanism is shown in Fig. 5(c). Both the photocatalytic hydrogenation and comparative experiment involve three steps: PLE adsorption, active hydrogen species (H-Pt) forming and attack of C≡C, and the product desorption. For the photocatalytic hydrogenation of PLE, the photogenerated electrons and holes are produced under UV light. Then the photogenerated electrons would migrate to the surface of Pt cocatalyst. CH₃OH as hydrogenation source and electron donor can be oxidized by photogenerated holes of TiO₂, and thus the H⁺ dissociates into the solution. Afterwards, active hydrogen species (H-Pt) are formed on the surface of Pt cocatalyst by reducing protons (H⁺) through photogenerated electrons, and PLE is hydrogenated by active hydrogen species (H-Pt). Finally, the formed STE will detach from the surface of catalyst. As reported by previous works [29,30], the increasing electron density of catalyst will cause the decreased adsorption strength of intermediate STE. Desorption of STE is favored, so the selectivity towards STE is enhanced. It should be pointed out that the electron density of Pt under photocatalytic hydrogenation process is more than the comparative experiment, because the photogenerated electrons transfer from the TiO₂ to the Pt cocatalyst under UV light. Thus we infer that the high electron

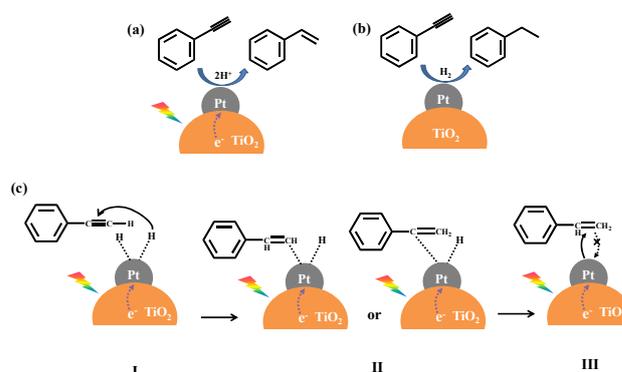


Fig. 5. (a) Schematic illustration of the photocatalytic hydrogenation of PLE to STE over the Pt/TiO₂ photocatalyst in the methanol suspension; (b) the comparative experiment over the Pt/TiO₂ with H₂ as hydrogenation source; (c) Proposed photocatalytic reaction mechanism for semi-hydrogenation of PLE over Pt/TiO₂.

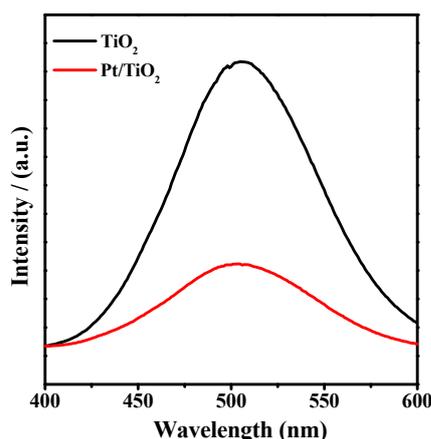


Fig. 6. PL spectra of pure TiO₂ and Pt/TiO₂ samples.

density of Pt cocatalyst is responsible for the enhanced selectivity towards STE in photocatalytic hydrogenation of PLE. But the low electron density of Pt free of light in comparative experiment isn't favored for the desorption of STE, and produces EBE (product of over-hydrogenation). Pt cocatalyst not only acts the reaction sites of PLE hydrogenation, but also can collect the photogenerated electrons from the conduction band of TiO₂ to extend the life time of photogenerated electrons. The PL spectra in Fig. 6 could help to understand the fate of the photogenerated electrons and holes in the semiconductor, in which an obvious fluorescence quenching of Pt/TiO₂ is observed compared with TiO₂. And the quenching of fluorescence indicates the transfer of photogenerated electrons from TiO₂ to Pt nanoparticles to reduce the recombination of photogenerated electrons and holes of TiO₂.

In summary, taking hydrogenation of PLE as the example, we show that Pt/TiO₂ photocatalyst can selectively hydrogenate alkynes to alkenes with high conversion rate under monochromatic light irradiation. Different alkynes are also successfully semi-hydrogenated to the corresponding alkenes. It is demonstrated that the photocatalytic hydrogenation approach outperforms the comparative experiment with H₂ as hydro-

genation source in the controllable hydrogenation extent. This work emphasizes the advantage of photocatalysis in the semi-hydrogenation of alkynes in a cleaner, safer, and highly selective manner, which may be useful for the design of highly selective hydrogenation systems in the alkyne conversion.

Acknowledgments

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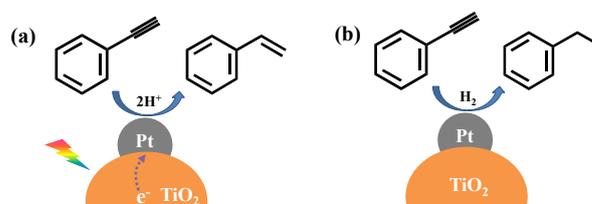
Graphical Abstract

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Unexpectedly selective hydrogenation of phenylacetylene to styrene on titania supported platinum photocatalyst under 385 nm monochromatic light irradiation

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Compared with the traditional catalytic hydrogenation of phenylacetylene using H₂ as hydrogenation source, photocatalytic phenylacetylene hydrogenation with methanol as hydrogenation source is a cleaner, safer, and highly selective manner.



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铂负载二氧化钛光催化剂在单波长下苯乙炔高选择性加氢

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摘要: 苯乙烯是一种非常重要的化工原料, 它是工业上生产聚苯乙烯、树脂和丁苯橡胶的重要单体, 而苯乙烯单体中经常含有苯乙炔杂质, 影响苯乙烯的聚合性能, 因此研究苯乙炔选择性加氢生成苯乙烯具有十分重要的工业意义. 传统的热催化苯乙炔加氢反应会用到易燃易爆的氢气, 引起操作的危险性, 因此, 开发具有环境友好型的加氢反应体系具有很重要的意义. 光催化加氢反应利用光生电子强的还原能力, 还原质子产生活性的氢物种加氢, 相较于传统的热催化使用氢气作为加氢源加氢, 能够在温和的条件下实现高选择性加氢. 基于此, 本文利用Pt/TiO₂作为光催化剂, 甲醇作为加氢源实现了在385 nm单波长光照下苯乙炔的高选择性加氢.

首先, 我们利用光沉积的方法将Pt负载在TiO₂的表面, 通过透射电子显微镜图像和紫外可见吸收光谱表征了负载在TiO₂表面Pt的颗粒分布和光学性质. 结果表明负载的Pt的颗粒大约在5 nm左右, Pt的负载改变了TiO₂在可见光区的吸收性能. XPS结果显示, 通过光沉积得到的Pt的价态为金属态和氧化态共存. 光催化苯乙炔加氢实验表明, Pt/TiO₂催化剂在室温常压条件下不仅具有高的苯乙炔光催化转化率, 当光照达8 h后, 苯乙炔完全转化, 而且在6 h之内苯乙烯选择性保持在91.3%, 具有高的苯乙烯选择性. 通过对负载的Pt的含量进行了优化, 筛选出当Pt的负载量为1 wt%时, 苯乙炔的转化率最高. 为了对比, 利用传统热催化的方法氢气作为加氢源进行了苯乙炔加氢实验, 结果发现, 使用氢气作为加氢源时, 虽然苯乙炔的转化率为100%, 但产物是过加氢的产物乙苯. 这主要是因为光催化反应过程中, TiO₂导带上的电子迁移至Pt颗粒上, 导致Pt的电子密度增加, Pt颗粒表面高的电子密度有利于加氢中间产物苯乙烯的脱附, 因此, 在光催化加氢过程中不会发生过加氢反应, 具有高的苯乙烯选择性. 同时, 扩展实验表明, Pt/TiO₂光催化剂对其他类型的炔烃加氢也具有高的选择性, 表明Pt/TiO₂光催化炔烃加氢具有普适性. 由此可见, 光催化炔烃加氢未来将成为一种环境友好而高效的方法.

关键词: 苯乙炔; 二氧化钛; 选择性加氢; 光催化; 铂

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