



Effects of tungsten oxide on the activity and thermal stability of a sulfate-derived titania supported platinum catalyst for propane oxidation

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Abstract

A Pt/WO₃/TiO₂ catalyst for propane oxidation was prepared by a stepwise wet impregnation method, and was aged at 800°C for 5 hr. Compared to the sulfate-derived titania supported catalyst, the introduction of tungsten oxide as stable Brønsted acid sites led to the formation of more metallic platinum active sites at the Pt/WO₃ interface. The dissociation of surface intermediates for propane oxidation was promoted on the WO₃-modified catalyst. This, as well as the inhibition effects of tungsten oxide on the sintering of anatase and the phase transformation to rutile, resulting in a high activity and thermal stability for the Pt/WO₃/TiO₂ catalyst.

Key words: platinum; acidic support; propane oxidation; infrared spectra; ageing

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Introduction

In the past decades, platinum-based propane oxidation catalysts for the reduction of pollutant emission have been widely studied (Hinz et al., 2001; Wu et al., 2001; Kim et al., 2010; Yazawa et al., 2002a; Corro et al., 2002, 2003). Their activities are found to depend strongly on the acidity of the supports. The oxidation of platinum is more effectively inhibited with increase in the acidity of the support materials (Yazawa et al., 2002b), and more metallic platinum shows higher activity for propane oxidation (Yoshida et al., 2003). Moreover, the adsorption of propane has been reported to be important for Pt/zeolite catalysts (Garetto et al., 2004).

Titania shows superior catalytic performance for the oxidation of CO (Alayon et al., 2009) and volatile organic compounds (Papaefthimiou et al., 1998a, 1998b; Zhang and He, 2007; Zhang et al., 2005a) compared to alumina when used as the support for platinum catalysts, due to the strong interaction between platinum and titania. A further enhancement in the propane oxidation activity of Pt/TiO₂ catalysts can be achieved by sulfation (Ruth et al., 2000) or by using TiO₂-SiO₂-WO₃ as the acidic support (Kobayashi et al., 2007). However, the low thermal stability of anatase titania and sulfates limits their application as stable platinum catalyst supports and additives when subjected to unexpectedly high temperatures.

In consideration of practical applications, acidic addi-

tives may have higher potential than the support material to control the oxidation-resistance of platinum catalysts. Tungsten oxide has been reported to be an effective promoter for Pt/Al₂O₃ catalysts in propane oxidation (Yazawa et al., 2002c; Yoshida et al., 2003; Men et al., 2009). In the present study, titania derived from titanyl sulfate which contained ca. 0.85 wt.% SO₃ was used as a reference support for Pt catalysts. Tungsten oxide was impregnated on the sulfated titania as an enhanced acidic support. The thermal stabilities of the catalysts were examined by treatment at 800°C for 5 hr. Infrared spectra were recorded in contact with NH₃, CO and C₃H₈ to explore the possible mechanism of WO₃ modification on the propane oxidation activity of Pt/TiO₂ catalysts.

1 Materials and methods

1.1 Catalyst preparation

WO₃/TiO₂ (WT) was synthesized by impregnating 5(NH₄)₂O·12WO₃·5H₂O (99%, Juneng, China) on Millenium DT-51 TiO₂ at a weight ratio of WO₃:TiO₂=15:85. The manufacturing process of TiO₂ used titanyl sulfate as an intermediate and left (1.25 ± 0.45) wt.% residual sulfate content (expressed as SO₃). The impregnated support was dried at 110°C overnight and calcined at 500°C for 3 hr.

The Pt catalysts were prepared by a similar method on the TiO₂ and WO₃/TiO₂ supports. The nominal loading of Pt was 1 wt.% with an aqueous solution of Pt(NO₃)₂ (Heraeus GmbH, Germany) used as the precursor.

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sor. The impregnated powders were subjected to drying at 110°C overnight and calcination at 500°C for 1 hr to obtain PT (Pt/TiO₂) and PWT (Pt/WO₃/TiO₂) catalysts. The prepared powders were treated at 800°C for 5 hr in a furnace to obtain the aged catalysts designated with a suffix of “-a”.

1.2 Catalyst characterization

Powder X-ray diffraction (XRD) patterns were measured with a Japan Science D/max-RB diffractometer employing Cu K α radiation ($\lambda = 0.15418$ nm). The X-ray patterns were recorded at 0.02° intervals in the range of $20^\circ \leq 2\theta \leq 80^\circ$ with a scanning velocity of 4°/min. The mean crystallite sizes of titania in the samples were calculated according to the Williamson-Hall equation.

The specific surface areas of the samples were measured using the N₂ adsorption isotherm at -196°C by the four-point Brunauer-Emmett-Teller (BET) method using an automatic surface analyzer (F-Sorb 3400, Gold APP Instrument, China). The samples were degassed at 200°C for 2 hr prior to the measurements.

Raman spectra were obtained with a LabRAM HR 800 (HORIBA Jobin Yvon, France) spectrometer at room temperature (RT) and atmospheric pressure. An argon ion laser beam with the wavelength of 488 nm was focused on a spot of 1 μ m in diameter.

Infrared (IR) spectra of ammonia adsorbed on the catalysts were recorded on a Nicolet 6700 FT-IR spectrometer (Thermo Scientific, USA) equipped with a MCT detector. The catalyst was purged with nitrogen at 500°C for 30 min. A gas mixture of 1000 ppm NH₃/N₂ was dosed in a diffuse reflectance IR cell at RT for 30 min, and then the catalyst was flushed with N₂. The spectra were collected by accumulating 32 scans at a resolution of 4 cm⁻¹.

IR spectra of CO adsorption were recorded as described above. After pretreatment in nitrogen at 500°C for 30 min, the catalyst was cooled to RT. The spectra were taken after exposing the catalyst to 1% CO/N₂ for 30 min and then purging with nitrogen.

IR spectra of propane adsorption/oxidation were performed on the same apparatus. After pretreatment in nitrogen at 500°C for 30 min, the catalyst was cooled to 200°C. The spectra were recorded after exposing the catalyst to 800 ppm C₃H₈/N₂ for 30 min and after the successive purging with 2% O₂/N₂ for 20 min.

1.3 Activity measurement

The activities of the catalysts for propane oxidation were tested in a fixed bed reactor made of stainless steel (i.d. 18 mm). Catalyst powders 0.2 g (diluted to 1 mL with silica pellets), sandwiched between two quartz wool layers, were inserted into the reactor. After a pretreatment at 500°C in N₂ for 30 min and cooling to RT, the reactor was heated to 400°C at a rate of 10°C/min. The gas mixture consisted of 800 ppm C₃H₈, 2% O₂ and N₂ in balance with a total flow rate of 1000 mL/min. The outlet gas concentrations were determined by a MultiGasTM 2300 IR spectrometer (MKS Instrument, USA).

2 Results and discussion

2.1 Solid properties

The XRD patterns of the fresh and aged catalysts are shown in Fig. 1. All the fresh catalysts exhibit the characteristic peaks of anatase titania. No diffraction peaks of crystalline WO₃ are observed for PWT, suggesting that WO₃ clusters are highly dispersed on the surface of titania due to the impregnation method adopted. The absence of any platinum peaks also indicates a high degree of the metal dispersion. No diffraction peaks of sulfates are observed, indicating the amorphous state of sulfates or their presence in the titania matrix. The BET surface area decreased slightly from 93 to 85 m²/g after the impregnation with tungsten oxide due to blocking of the support pores by the additive.

The thermal treatment at 800°C resulted in a partial transformation of anatase titania to the rutile phase, which was effectively prevented by WO₃ modification. Meanwhile, the sintering of anatase titania in PWT-a was inhibited to some extent, with a smaller crystallite size (17 nm) compared to that in PT-a (20 nm). This maintained a relatively higher surface area (26 m²/g) for PWT-a compared to PT-a (7 m²/g). The diffraction peaks of metallic Pt⁰ are observed for both the aged catalysts. However, Pt seemed to suffer a more severe sintering without the WO₃ modification as indicated by the sharper Pt (200) peak in the pattern for PT-a in the inner figure. That is, the WO₃/TiO₂-supported catalyst maintained a higher level of platinum dispersion after ageing, which may be related to the stabilizing effect of tungsten oxide on the high-surface-area anatase titania.

It can be seen from the XRD pattern of PWT-a that crystalline WO₃ forms due to sintering of tungsten oxide. To further illuminate this transformation of WO_x to WO₃, the Raman spectra of the W-containing catalysts are shown in the 700–1100 cm⁻¹ region in Fig. 2. The spectrum of PWT reveals a Raman band at 980 cm⁻¹ which is a characteristic of W=O vibrations arising from polytungstates. This is similar to the spectra of WO_x species strongly

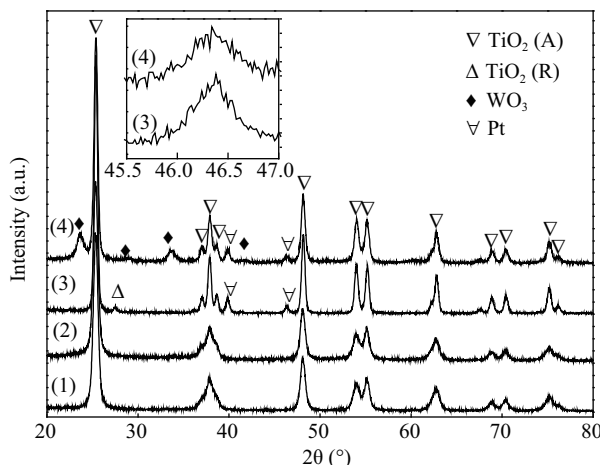


Fig. 1 XRD patterns of PT (line 1), PWT (line 2), PT-a (line 3), and PWT-a catalysts (line 4). PT: Pt/TiO₂; PT-a: Pt/TiO₂ after thermal aging; PWT: Pt/WO₃/TiO₂; PWT-a: Pt/WO₃/TiO₂ after thermal aging.

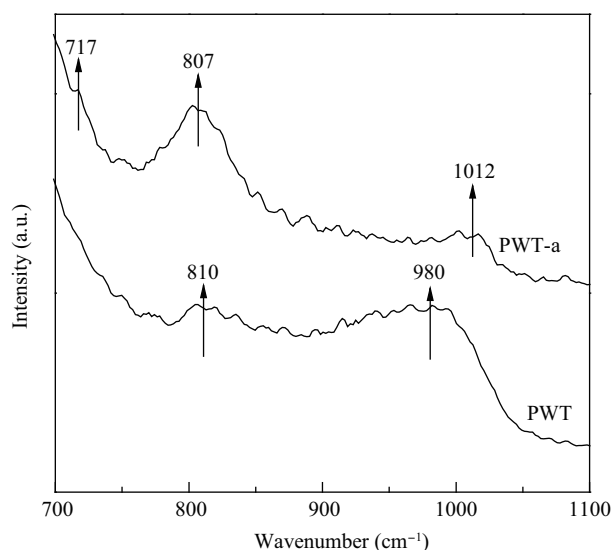


Fig. 2 Raman spectra of the fresh and aged Pt/WO₃/TiO₂ catalysts.

bonded with Al₂O₃ (Contreras et al., 2009), and suggests that the W^{δ+} species are well dispersed and isolated on TiO₂. A small band at 810 cm⁻¹ is found which is characteristic of crystalline octahedral WO₃ species, implying the occurrence of sintering of tungsten oxide locally. After the thermal treatment, Raman bands corresponding to crystalline WO₃ appear at 807 and 717 cm⁻¹, which are assigned to W–O stretching and W–O bending modes, respectively. The W=O Raman band decreases in intensity and shifts to higher wavenumber at 1012 cm⁻¹ (Barton et al., 1999), which may be related to the loss of highly dispersed tungsten oxide clusters and the weakening of the interaction between tungsten oxide and titania.

2.2 Catalytic activity

The propane oxidation activities of the catalysts are shown in Fig. 3. It can be seen that WO₃ modification leads to a significant improvement in the activities of both fresh and aged catalysts. The light-off temperature (T_{50}) follows the order of PWT (216°C) < PT (233°C) < PWT-a (254°C) < PT-a (311°C). The shift in T_{50} to lower temperatures for Pt/TiO₂ catalyst due to WO₃ modification is 17 and 57°C before and after ageing respectively. Furthermore, the maximal propane conversion only reaches 80% over

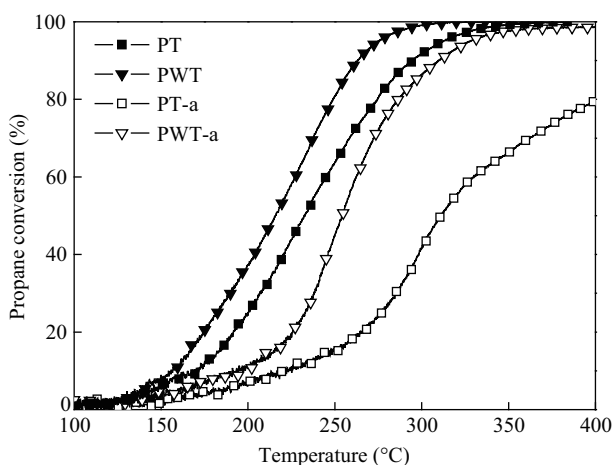


Fig. 3 Propane oxidation activities of the catalysts.

PT-a at 400°C. These differences demonstrate an enhanced catalytic activity and improved thermal stability for the WO₃-modified catalyst.

2.3 NH₃ adsorption

In order to assess the effect of the WO₃ modification on the acidity of the support, the IR spectra obtained after NH₃ adsorption on the catalysts are shown in Fig. 4. Typical bands at 3400–3348, 3258 and 3166 cm⁻¹ are assigned to $\nu_{as}(N-H)$, $\nu_s(N-H)$ and $\delta_{as}(NH)$ of Lewis-bound ammonia, respectively. Broad bands appear in the range 3100–2600 cm⁻¹ due to Brønsted-bound ammonia (Fabrizioli et al., 2002). The bands at 1676 and 1456 cm⁻¹ are characteristic of asymmetric and symmetric NH₄ stretching (Vargas et al., 2007; Jung and Grange, 2000). The bands corresponding to Lewis- and Brønsted-bound ammonia, which are suggested to be coordinated to sulfates and tungsten oxide species, are clearly seen on PT and become even stronger on PWT. The presence of sulfates on PT is evidenced by the bands at 1277, 1212 and 1060 cm⁻¹ corresponding to sulfate, sulfite and surface sulfite/sulfate species, respectively (Hinz et al., 2001). The pronounced negative adsorption band at ca. 1360 cm⁻¹ is assigned to the $\nu(S=O)$ vibration of volatile sulfate surface species originating from the synthesis procedure of the titania support (Österlund et al., 2009). These bands decrease in intensity on PWT due to the surface coverage of tungsten oxide. Instead, a band at 996 cm⁻¹ assigned to the W=O stretching of highly dispersed WO₃ clusters appears on this catalyst, which correlates with the Raman spectrum of PWT. Thus, the sulfate-derived titania-supported fresh catalyst shows moderate Lewis and Brønsted acidities, which can be strengthened by the introduction of WO₃.

After the ageing treatment, the bands assigned to Brønsted-bound ammonia disappear and those assigned to Lewis-bound ammonia decrease sharply in intensity on PT-a. This is associated with the decomposition of sulfates during calcination at 800°C as evidenced by the disappearance of the bands corresponding to sulfates. The band at 996 cm⁻¹ decreases sharply in intensity on PWT-a, which is consistent with the Raman spectrum and indicates

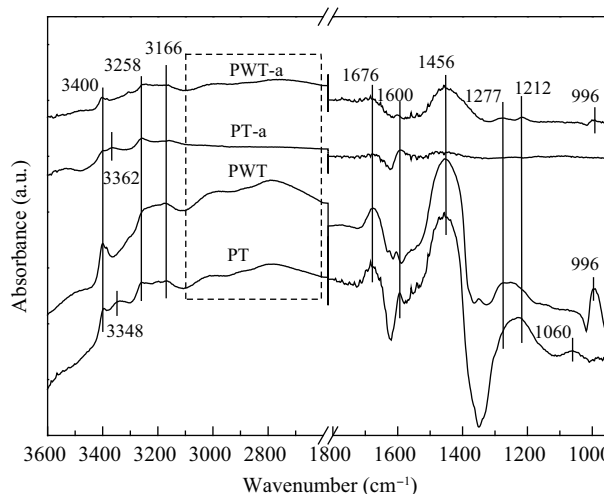


Fig. 4 IR spectra of the species adsorbed on catalysts in contact with ammonia at RT.

the sintering of tungsten oxide. It is noted that weak bands corresponding to sulfates exist on this catalyst, implying that the introduction of tungsten oxide also contributes to the retention of sulfates. Thus, the residual WO_3 clusters and sulfates endow PWT-a with certain Lewis and especially Brønsted acidities. In this sense, tungsten oxide is more thermally stable than sulfates as an acidic additive for Pt/TiO₂ catalysts.

2.4 CO adsorption

IR measurement of adsorbed CO is one of the most informative and simple techniques used for the characterization of supported noble metal catalysts in the metallic state. As shown in Fig. 5, a band at 2070 cm⁻¹ and a shoulder at 2117 cm⁻¹ are observed on PT, which are assigned to linear CO adsorbed on metallic platinum (Pt⁰) and partially oxidized platinum (Pt^{δ+}) sites, respectively (Ivanova et al., 2007). These bands shift to 2085 and 2128 cm⁻¹ for PWT, respectively, with the increase in the acidity of the support (Yazawa et al., 2002a). Thus, platinum on the strongly acidic WO₃/TiO₂ support is more electron-deficient than that on the moderately acidic TiO₂ support prepared by the sulfate route. The band assigned to CO coordinated to Pt⁰ sites becomes much more intense on PWT, reflecting a large number of exposed platinum atoms.

After ageing, the CO stretching bands disappear almost completely on PT-a, while the band associated with CO-Pt⁰ exists at a low intensity on PWT-a. The CO band position and intensity are sensitive to various factors, such as platinum dispersion, oxidation state of platinum and charge transfer in the vicinity of Pt adsorption sites (Zafeiratos et al., 2005). Since the XRD patterns have evidenced the existence of metallic Pt crystallites on the aged catalysts, the disappearance of the CO band on PT-a can be ascribed to the sintering of Pt as well as the encapsulation of Pt by the low-surface-area titania under the oxidizing atmosphere (Zhang et al., 2005b), which are responsible for the thermal deactivation of the Pt/TiO₂ catalyst. The residual CO-Pt⁰ adsorption band on PWT-a indicates a relatively high Pt dispersion after ageing, which may be

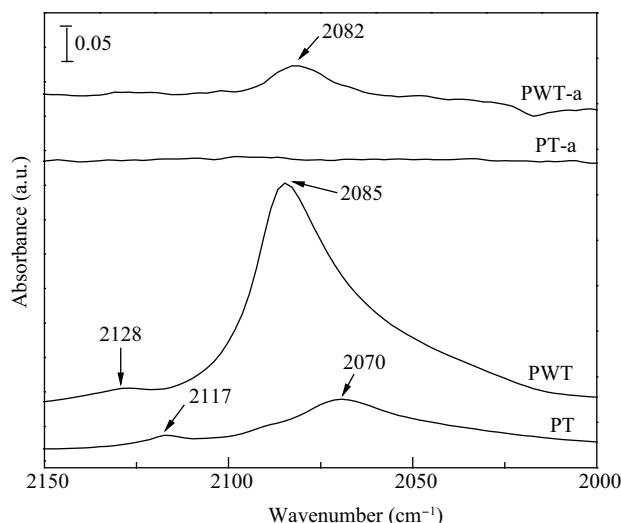


Fig. 5 IR spectra of the species adsorbed on catalysts in contact with CO at RT.

related to the relatively high surface area of the catalyst and interaction between platinum and the acidic support.

2.5 Propane adsorption

Figure 6 shows the IR spectra of the adsorption of propane on the fresh catalysts, to gain insight into the nature of the surface species formed in the course of the catalytic reaction without any oxidizing reactants. The oxygen supply arises from the surface-adsorbed and lattice oxygen on the catalysts. A band at 2975 cm⁻¹, which is characteristic of the C–H vibration of gaseous propane (Hinz et al., 2001; Shee and Deo, 2009), is observed after propane adsorption on both catalysts. The propane adsorption ability of the Pt/TiO₂ catalyst seems to be enhanced to some extent by WO₃ modification, which may be associated with the presence of more platinum species in the metallic state due to interaction with the more strongly acidic additive, despite the fact that the two fresh catalysts exhibit similar surface areas. However, the enhancement of propane uptake is not drastic. Thus a contributing oxidation pathway involving propane adsorbing on the metal-oxide interface region and reacting with oxygen spilled-over from platinum (Garetto et al., 2004) is very limited in addition to the main oxidation pathway on platinum.

According to previous publications (Shee and Deo, 2009; Koziej et al., 2005; van der Meulen et al., 2007; Ermini et al., 2000), propene (993 and 903 cm⁻¹), acetone (1688, 1421, 1245 and 1140 cm⁻¹) and formate (1594, 1557, 1336 and 1290 cm⁻¹) are major adsorbed species on PT after purging with C₃H₈. These adsorption bands decrease in intensity on PWT, and the importance of acetate (1521 and 1447 cm⁻¹) is enhanced. Meanwhile, the band at 2056 cm⁻¹ assigned to linearly adsorbed CO on metallic platinum is sharpened pronouncedly on the WO₃-modified catalyst. That is, less oxygenated species but more complete product (CO) is produced on PWT, revealing a higher reactivity for the decomposition of surface intermediates. The sharp band at 1630 cm⁻¹ on PT is due to the $\delta(\text{HOH})$ vibration of adsorbed water, and the negative band at 1380 cm⁻¹ is related to the $\nu(\text{S}=\text{O})$ vibration of volatile sulfate surface species. There have

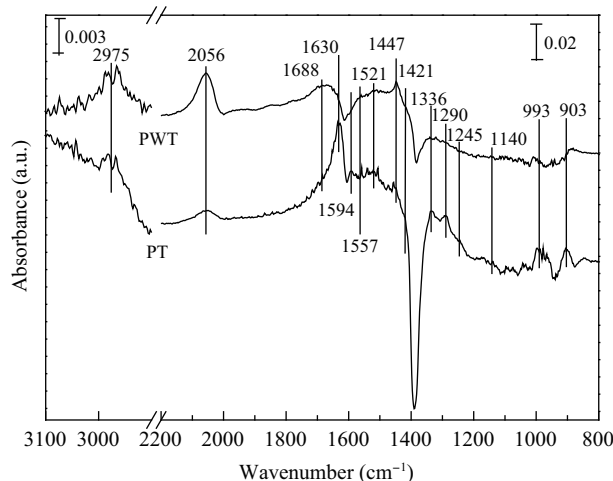


Fig. 6 IR spectra of the species adsorbed on catalysts in contact with propane at 200°C.

been several reaction pathways presented in the literature (Koziej et al., 2005), and the oxidation of strongly bound intermediate surface species has been found to be rate-determining for propane oxidation in many studies (van der Meulen et al., 2007). In our investigation we could not obtain any direct proofs for specific reaction pathways such as oxidative dehydrogenation of propane to propene or oxidative addition of surface oxygen resulting in acetone, formate and acetate. However, it can be seen that the oxidation of these intermediates clearly limits the total oxidation of propane on Pt/TiO₂, and the introduction of tungsten oxide promotes the decomposition of surface intermediates (propene, acetone and formate) on more active metallic platinum sites via a strong interaction between the noble metal and the acidic additive.

3 Conclusions

A platinum catalyst supported on sulfate-derived titania was modified by impregnation with tungsten oxide. The propane oxidation activity of the fresh catalyst was found to be promoted by the introduction of WO₃. The promotion effect of the acidic additive was limited since the sulfates distributed in the titania matrix could themselves act as Brønsted and Lewis acid sites. The WO₃-modified catalyst was found to show a much higher thermal stability after thermal ageing at 800°C. Tungsten oxide was suggested to play at least three important roles: (1) stabilization of the titania support in the high-surface-area anatase phase; (2) maintaining the metallic state of platinum by providing more stable Brønsted acid sites; and (3) formation of more active sites at the Pt/WO₃ interface to facilitate the dissociation of surface intermediates for propane oxidation.

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