

Effects of V_2O_5 and WO_3 loadings on the catalytic performance of V_2O_5 - WO_3 / TiO_2 catalyst for SCR of NO with NH_3

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Abstract

V_2O_5 - WO_3 / TiO_2 catalysts were fabricated by a simple impregnation method. Effects of V_2O_5 and WO_3 loadings on the catalytic performance of V_2O_5 - WO_3 / TiO_2 catalyst for selective catalytic reduction (SCR) of NO with NH_3 were investigated. Morphology and structure of the V_2O_5 - WO_3 / TiO_2 catalysts were characterised by XRD, SEM, XPS, and N_2 adsorption techniques. The XRD patterns of the V_2O_5 - WO_3 / TiO_2 catalyst are indexed to anatase- TiO_2 . XPS spectra analysis confirms that V, Ti, W and O species exist on the surface of V_2O_5 - WO_3 / TiO_2 catalyst. V_2O_5 species are the main active sites in the process of SCR reaction. Increasing V_2O_5 loading in the V_2O_5 - WO_3 / TiO_2 catalysts can improve their catalytic performance. Exceeding 2 wt%, the catalytic performance of V_2O_5 - WO_3 / TiO_2 catalyst begins to decline because high V_2O_5 loading on TiO_2 speeds up the growth of anatase grains, which leads to the loss of catalytic activity. Appropriate WO_3 species can significantly improve the catalytic performance of V_2O_5 - WO_3 / TiO_2 catalysts. However, as the WO_3 loadings reaches 6 wt%, NO conversion decreases instead.

Keywords: V_2O_5 - WO_3 / TiO_2 catalyst; SCR; Impregnation methods; Chemisorbed oxygen

1. Introduction

Nitrogen oxides (NO_x), as major atmospheric pollutants (Yu *et al.*, 2013; Liu *et al.*, 2014), have caused a series of environmental issues, such as ozone depletion, acid rain, photochemical smog, particulate matters (PM_{2.5}) transformation, greenhouse effect etc (Skalska *et al.*, 2010; Kompio *et al.*, 2012; Yang *et al.*, 2014; Huang *et al.*, 2015; Ma *et al.*, 2015). In recent years, with the significant growth of environmental awareness, the environmental problems arising from nitrogen oxides (NO_x) have been paid more attention. Facing the rising social dissatisfaction with the state of the environment, Governments, especially in Europe, Japan, the United States and China (Gao *et al.*, 2013; Jiang *et al.*, 2016), have enacted a series of stringent emission regulations. Also, many researchers develop various promising technologies, such as NO_x storage and reduction (NSR), the lean NO_x trap (LNT), and selective catalytic reduction (SCR) of NO_x with ammonia, to solve

those issues. Among these methods, NSR usually requires a large amount of fuel to create a reductive atmosphere. LNT is relatively simple and efficient, but it needs expensive platinum group metal (PGM) (Herrerros *et al.*, 2014; Gu *et al.*, 2015; Raptotasios *et al.*, 2015; Seo *et al.*, 2015; Wang *et al.*, 2015). SCR is considered to be the most efficient technology due to its high efficiency, selectivity and economy, and becomes the leading NO_x control strategy for decades (Shakya *et al.*, 2015; Zhang *et al.*, 2015).

Many catalysts have been reported to be adopted for the SCR reaction, such as loaded V, Cu, Fe and Mn species over TiO_2 , Al_2O_3 , AC and zeolites supports (Leistner and Olsson, 2015; Usbertia *et al.*, 2015; Romero-Sáez *et al.*, 2016). The most important catalyst currently used for SCR is based on V_2O_5 - WO_3 / TiO_2 catalyst because it has a relatively high activity and N_2 selectivity at 300-400°C. Up to now, many works on the preparation of V_2O_5 - WO_3 / TiO_2 catalysts and their application on the selective catalytic reduction (SCR) of NO with NH_3 were reported. Rodella and Mastelaro investigated the influence of the vanadium load and calcination temperature on the structural characteristics of the V_2O_5 / TiO_2 system obtained by the sol-gel method, and proved it is useful to modify the structure of the V_2O_5 / TiO_2 system by adjusting different amounts of vanadium and calcination temperatures (Rodella and Mastelaro, 2003). Kobayashi and Hagi reported their work on the V_2O_5 - WO_3 catalysts loaded on the Ti-rich TiO_2 - SiO_2 - SO_4^{2-} by the coprecipitation method, and clarified the effect of active components and supports on activities in the selective catalytic reduction of NO by NH_3 (Kobayashi and Hagi, 2006). Djerad *et al.* synthesized V_2O_5 - WO_3 / TiO_2 catalysts by the sol-gel method, and found the catalytic behavior of V_2O_5 - WO_3 / TiO_2 catalyst is strongly dependent on V_2O_5 content (Djerad *et al.*, 2004). Dong *et al.*, investigated the effect of the pH value of vanadium precursor solution on the catalytic performance of V_2O_5 - WO_3 / TiO_2 catalysts through wet impregnation, and indicated that high de- NO_x activity of the V_2O_5 - WO_3 / TiO_2 catalysts can be obtained by enhancing the precursor solution acidity in the preparation process (Dong *et al.*, 2014). Pang *et al.*, demonstrated that ultrasound-assisted impregnation method can promote the strong interaction between the V_2O_5 and TiO_2 - WO_3 support and stabilize V_2O_5 in the reduced state (Pang *et al.*,

2014). Zhang *et al.*, fabricated the V_2O_5 - WO_3 / TiO_2 catalyst by a sol-gel method, and investigate the interaction of V, W and Ti species for the improvement of the catalytic activity in the SCR reaction. They found that WO_3 could interact with TiO_2 to improve the electrons transfer, and the WO_3 hybridization with V_2O_5 could also improve the reducibility and formation of reduced V_2O_5 species (Zhang *et al.*, 2015). These previous studies have shown that the catalytic properties of V_2O_5 - WO_3 / TiO_2 catalysts are markedly influenced by some parameters, such as preparation methods and conditions, concentration of the active component etc. In this study, we synthesized V_2O_5 - WO_3 / TiO_2 catalysts using a simple impregnation method, investigated the morphology and structure characteristics of the V_2O_5 - WO_3 / TiO_2 catalysts by XRD, SEM, XPS, and N_2 adsorption techniques, and further illustrated the effects of V_2O_5 and WO_3 loadings on the catalytic performance of V_2O_5 - WO_3 / TiO_2 catalyst in the selective catalytic reduction (SCR) of NO with NH_3 .

2. Experimental

2.1. Preparation of V_2O_5 - WO_3 / TiO_2 catalyst

The V_2O_5 - WO_3 / TiO_2 catalyst was prepared by impregnation method. Sodium Tungstate Dihydrate (Na_2WO_4) (WO_3 loadings were 1 wt%, 3 wt%, and 6 wt%, respectively) and 0.96 g oxalic acid ($H_2C_2O_4$) were dissolved in 6.8 mL deionized water under magnetic stirring until they completely dissolved. Then 8 g commercial anatase TiO_2 powder was added into the above solution under stirring for 2 h at room temperature. After that, the above mixture was dried at 80 °C for 12 h, and then it was grinded and subsequently calcined at 450 °C for 4h. Ammonium metavanadate (NH_4VO_3) (V_2O_5 loadings were 0.5 wt%, 1.5 wt%, and 2 wt %, respectively) and 0.248 g oxalic acid were completely dissolved in 6.8 mL deionized water under magnetic stirring at room temperature. After TiO_2 - WO_3 powder was introduced in the solution under stirring for 2 h, the above mixture was dried at 80 °C for 12 h. Then it was grinded and subsequently calcined at 450 °C for 4 h. Finally, the V_2O_5 - WO_3 / TiO_2 powder was obtained, which was referred as $V_x\%TiW_y\%$. For example, V1.5%TiW6% meant the catalyst with 1.5 wt% V_2O_5 and 6 wt% WO_3 loadings.

2.2. Catalytic activity measurement

The activity measurements were carried out in a fixed-bed quartz reactor which contained 4.00 g catalysts of 20 mesh and 1.15 g quartz sand (another 1.15 g quartz sand to control air velocity). The feed gas mixture contained 0.05 vol % NO, 0.08 vol % NH_3 , 2.5 vol% O_2 and balanced N_2 at a flow rate of 330 mL/min controlled by mass flow meters. The reaction temperature was increased to 550 °C in a heating rate of 5 °C /min. The NO conversion was calculated by testing the NO concentration at the inlet and outlet of reactor according to China National Standard (GB/T 8969-1988).

2.3. Characterization

X-ray diffraction (XRD) patterns of V_2O_5 - WO_3 / TiO_2 catalysts were recorded using a D/Max-2400 diffractometer (Cu $K\alpha$

radiation, $\lambda = 1.54055 \text{ \AA}$) in a range of diffraction angle 2θ from 10 ° to 80 ° to analyze the diffraction peaks of V_2O_5 - WO_3 / TiO_2 catalysts. The pore structure properties of V_2O_5 - WO_3 / TiO_2 catalysts was characterized by using a Quantachrome Autosorb-iQ automated gas adsorption system with liquid nitrogen (at 77 K). The morphology of V_2O_5 - WO_3 / TiO_2 catalysts was observed by a Philips XL30 FEG scanning electron microscope (SEM). X-ray photoelectron spectroscopy (XPS) of V_2O_5 - WO_3 / TiO_2 catalysts was carried out on a Thermo Scientific ESCALAB 250 spectrometer with a monochromation Al $K\alpha$ source.

3. Results and discussion

3.1. Effect of V_2O_5 loadings on the catalytic performance of V_2O_5 - WO_3 / TiO_2 catalyst

Fig. 1 shows the typical XRD patterns of V_2O_5 - WO_3 / TiO_2 catalysts at different V_2O_5 loadings. All the diffraction peaks ($2\theta=25.44^\circ$, 38.10° , 48.17° , 54.38° , 55.13° , 62.75° , 70.48° , 75.30°) are indexed to anatase- TiO_2 (PDF#21-1272) (Zhang *et al.*, 2015). In addition, we also notice that the crystalline phase of V_2O_5 is not detected in all the XRD patterns, which may be attributed to low amounts of V_2O_5 in these catalysts, and thus no obvious diffraction peaks are observed. Furthermore, there are almost no significant differences on diffraction peaks with the increase of V_2O_5 loadings. Therefore, we can conclude that the particle size and crystal phase have little change by V_2O_5 loadings.

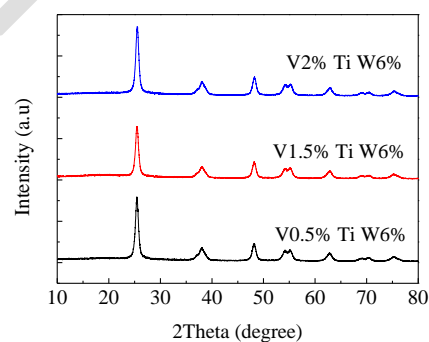


Figure 1. XRD patterns of V_2O_5 - WO_3 / TiO_2 catalyst at different V_2O_5 loadings

Fig. 2 depicts the high-resolution spectra of V2p, W4f, Ti2p and O1s, which suggests that these species exist on the surface of V_2O_5 - WO_3 / TiO_2 catalyst. As shown in Fig. 2a, three kinds of V species can be distinguished in the V2p spectrum. The binding energies at 514.88 eV, 516.08 eV and 516.98 eV are attributed to $V^{3+}2p_{2/3}$, $V^{4+}2p_{2/3}$ and $V^{5+}2p_{2/3}$, respectively, and V mainly exists as the V^{+5} oxidation state on the V_2O_5 - WO_3 / TiO_2 catalyst (Li *et al.*, 2015). Fig. 2b presents the W4f_{7/2} and W4f_{5/2} peaks in the XPS spectra, which are located at 36.18 eV and 37.48 eV, respectively, indicating the W^{6+} oxidation state. Fig. 2c shows the XPS spectra for Ti2p of V_2O_5 - WO_3 / TiO_2 catalyst. The two peaks at 464.18 and 458.58 eV are ascribed to Ti2p_{1/2} and Ti2p_{3/2}, respectively, suggesting that Ti exists as the Ti^{+4} state.

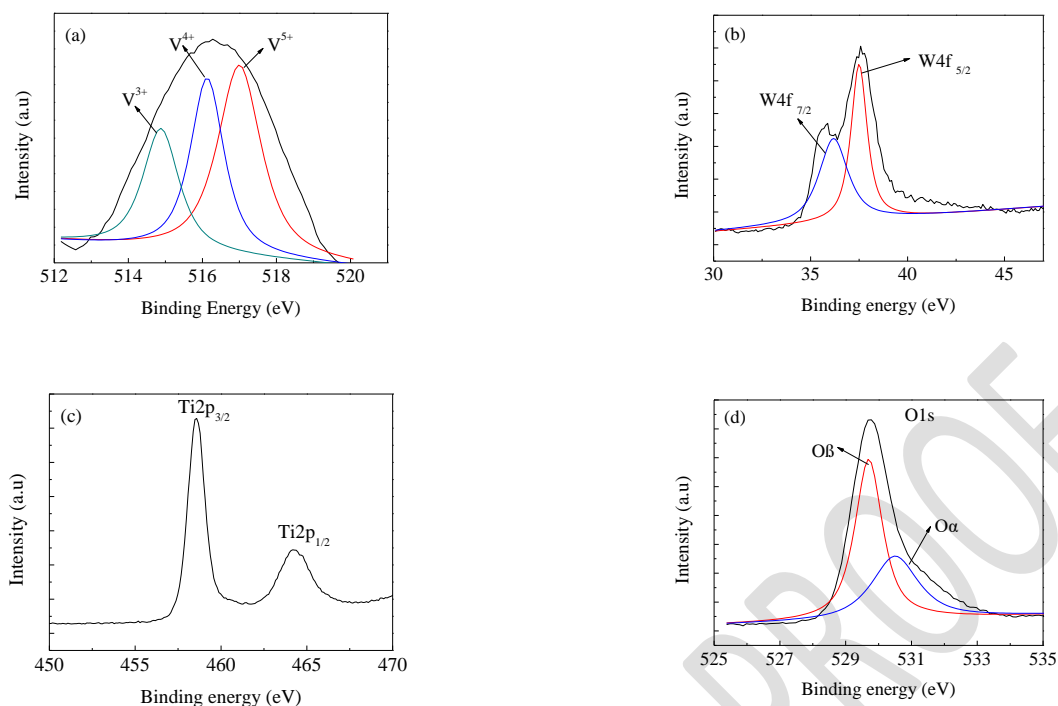


Figure 2. XPS spectra of V_2O_5 - WO_3 / TiO_2 catalysts: (a) V2p spectrum, (b) W4f spectrum, (c) Ti2p spectrum, and (d) O1s spectrum

Fig. 2d shows the O1s peaks of V_2O_5 - WO_3 / TiO_2 catalyst which can be fitted into two peaks. The binding energy at 529.68 eV 530.48 eV are referred to as the lattice oxygen ($O\beta$) and the chemisorbed surface oxygen ($O\alpha$), respectively (Ref 24). Generally, the chemisorbed surface oxygen ($O\alpha$) has a significant effect on the deep oxidation reactions of reducing substances. Therefore, the presence of the chemisorbed surface oxygen ($O\alpha$) will be beneficial to the SCR reaction. Fig. 3 is the typical SEM images of as-prepared V_2O_5 - WO_3 / TiO_2 catalysts at different V_2O_5 loadings (0.5 wt%, 1.5 wt%, and 2 wt%). The particles of V_2O_5 - WO_3 / TiO_2 catalysts show the sphere-like morphology with ranging from 30 to 50 nm. These spherical particles condense to form agglomerates. There are no apparent differences between the V_2O_5 - WO_3 / TiO_2 catalysts with 0.5 wt% and 1.5 wt% V_2O_5 loadings. As for 2 wt% V_2O_5 loading, the aggregations of catalytic particles are slightly serious.

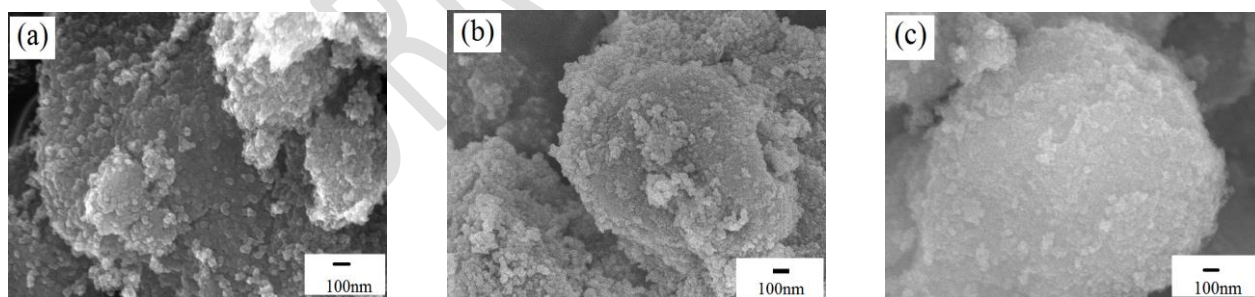


Figure 3. SEM images of V_2O_5 - WO_3 / TiO_2 catalysts: (a) V0.5TiW6%, (b) V1.5TiW6%, (c) V2%TiW6%

Fig. 4 shows NO conversion for the V_2O_5 - WO_3 / TiO_2 catalysts at different V_2O_5 loadings as a function of temperature from 100 to 550 °C. Obviously, the NO conversion for all the prepared catalysts increases significantly with increasing reaction temperature below 300 °C, and then reaches a steady state. When the reaction temperature is above 400 °C, the NO conversion begins to decrease. Among these catalysts, the two catalysts without V_2O_5 loadings (TiW1% and TiW6%) demonstrate low catalytic activity and narrow active windows. When V_2O_5 species are loaded, the catalytic performance of the catalysts is significantly

improved, and the maximal catalytic activity and widest active window (250 °C~500 °C) are achieved at 1.5 wt% of V_2O_5 loadings. All those imply that V_2O_5 species are the active sites on the V_2O_5 - WO_3 / TiO_2 catalyst because they can decrease the activation energy of SCR reaction (Amiridis and Solar, 1996; Busca *et al.*, 1998). However, further increasing V_2O_5 loading to 2 wt%, the catalytic performance of the catalysts declines instead. The phenomenon may be attributed to that high V_2O_5 loadings on TiO_2 speed up the growth of anatase- TiO_2 grains, which leads to the loss of catalytic activity (Wang *et al.*, 2016).

Therefore, the V_2O_5 loading of 1.5 wt% is recommended for NO conversion by V_2O_5 - WO_3 / TiO_2 catalyst.

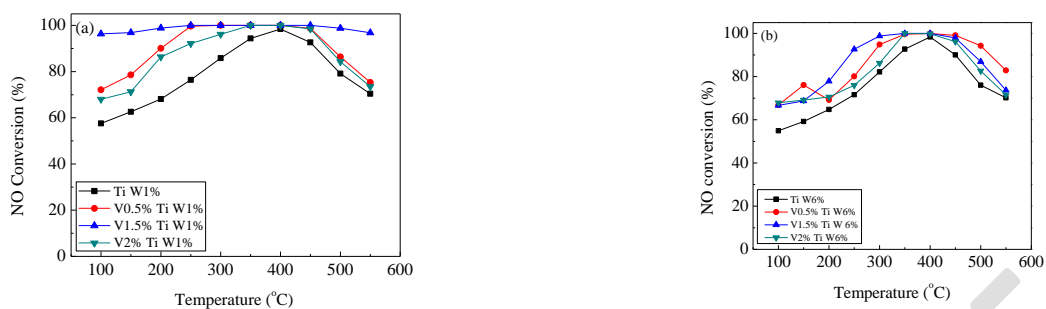


Figure 4. NO conversion for the V_2O_5 - WO_3 / TiO_2 catalysts with (a) 1 % WO_3 loading, (b) 6 % WO_3 loadings at different V_2O_5 loadings

3.2. Effect of WO_3 loadings on the catalytic performance of V_2O_5 - WO_3 / TiO_2 catalyst

Fig. 5 provides the XRD patterns of V_2O_5 - WO_3 / TiO_2 catalysts at different WO_3 loadings. Similarly, only diffraction peaks assigned to anatase- TiO_2 (PDF#21-1272) are observed, and diffraction peaks indexed to WO_3 are also not detected in the XRD patterns due to low WO_3 amount. Besides, there are no significant change on diffraction peak with the increase of WO_3 loadings, which implies that the particle size and crystal phase have little change by changing the WO_3 loadings. Fig. 6 shows the XPS spectra of W4f, V2p, Ti2p and O1s, which approves that these species exist on the surface of V_2O_5 - WO_3 / TiO_2 catalyst.

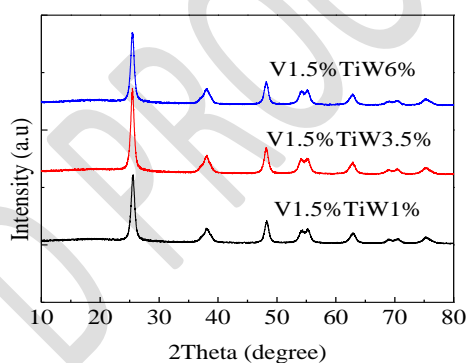


Figure 5. XRD patterns of V_2O_5 - WO_3 / TiO_2 catalyst at different WO_3 loadings

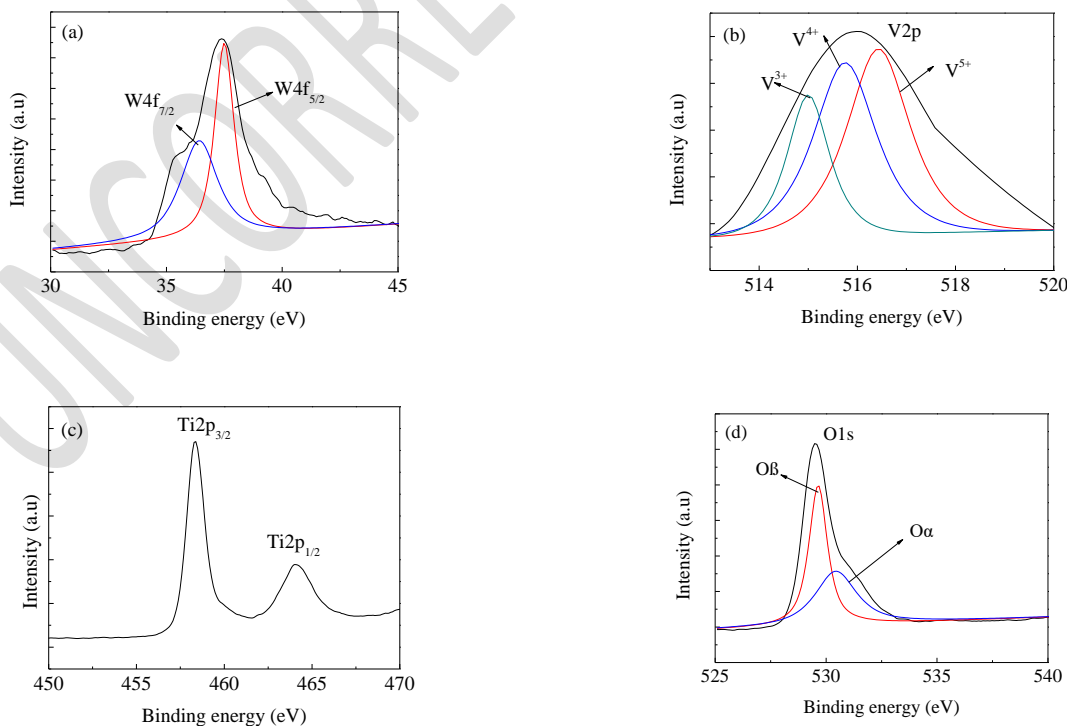


Figure 6. XPS spectra of V_2O_5 - WO_3 / TiO_2 catalyst: (a) W4f spectrum, (b) V2p spectrum, (c) Ti2p spectrum, (d) O1s spectrum

As shown in Fig. 6a, the binding energies at 37.48 and 36.38 eV are attributed to $W^{6+}4f_{5/2}$ and $W^{6+}4f_{7/2}$, respectively. Fig. 6b presents the information that three kinds of surface V species can be distinguished in the V2p spectrum. It is clear that the binding energies at 514.98, 515.78 and 516.48 eV are attributed to $V^{3+}2p_{2/3}$, $V^{4+}2p_{2/3}$ and $V^{5+}2p_{2/3}$, respectively, and V^{5+} state mainly exists on the V_2O_5 - WO_3 /TiO₂ catalyst. The two peaks at 464.08 and 458.38 eV are ascribed to $Ti^{4+}2p_{1/2}$ and $Ti^{4+}2p_{3/2}$, respectively (Fig. 6c). Fig. 6d shows the O1s peaks of V_2O_5 - WO_3 /TiO₂ catalyst which can be fitted into two peaks. The binding energy at 529.68 eV is referred as the lattice oxygen ($O\beta$) while at 530.48eV is referred to as the chemisorbed surface oxygen ($O\alpha$). As we known, surface chemisorbed oxygen has been reported to be the most active oxygen and plays an important role in oxidation reactions. Therefore, high ratio of $O\alpha/(O\alpha+O\beta)$ on the catalyst surface may also be related

to a high SCR activity (Chen *et al.*, 2011). As shown in Fig. 6d and Fig. 2d, the ratio of $O\alpha/(O\alpha+O\beta)$ of V1.5%TiW3.5% is higher than that in V1.5%TiW6% which indicates that V1.5%TiW3.5% has a better catalytic performance than V1.5%TiW6%. Fig. 7 shows the SEM images of V_2O_5 - WO_3 /TiO₂ catalysts fabricated at different WO_3 loadings (1 wt%, 3.5 wt%, and 6 wt%). The spherical catalytic particles are obviously agglomerated from the SEM images, which sizes are uniform. There are no apparent differences between the three V_2O_5 - WO_3 /TiO₂ catalysts. Fig. 8 presents the adsorption-desorption isotherms of V_2O_5 - WO_3 /TiO₂ catalyst, which demonstrates typical type IV curve with the hysteresis loop. The BET surface area is calculated to be 30.07 m²/g, which shows a facilitation effect toward the SCR reaction.

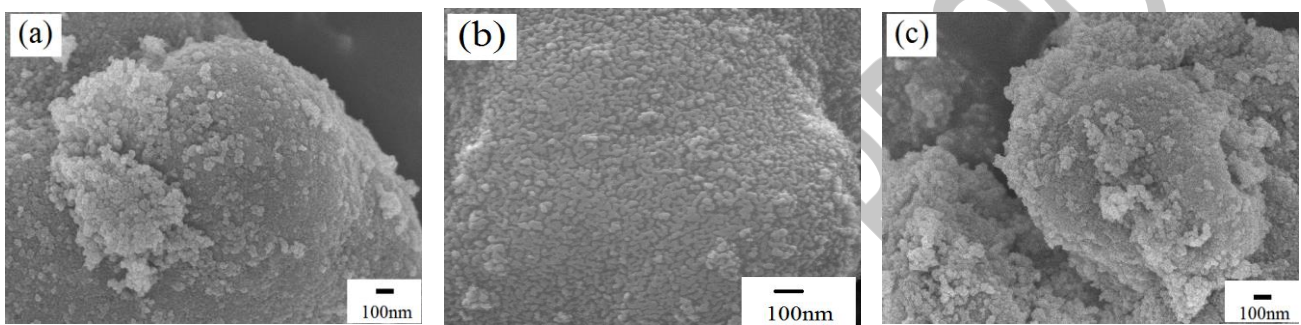


Figure 7. SEM images of V_2O_5 - WO_3 /TiO₂ catalyst: (a) V1.5%TiW1%, (b) V1.5%TiW3.5%, (c) V1.5%TiW6%

Fig. 9 shows NO conversion for the V_2O_5 - WO_3 /TiO₂ catalysts at different WO_3 loadings as a function of temperature from 100 to 550 °C. When the catalyst without WO_3 loading (V1.5%Ti) is adopted, the NO conversion is only 70.86% at 200 °C, and decreases significantly above 400 °C, which indicates its low catalytic activity and narrow temperature window. When the WO_3 loadings in the catalyst are 1 wt% and 3.5 wt%, the NO conversion of the prepared catalysts increase to 98.86 % and 98.53 % at 200 °C, and almost no NO can be detected above 250 °C (250–450 °C). This is because that the WO_3 species improve the electrons transfer of the V_2O_5 - WO_3 /TiO₂ catalyst, which results in the increase of the superoxide ions, prompting the SCR reaction. Increasing WO_3 loadings in the catalyst to 6 wt%, the NO conversion increases with the increase of reaction temperature below 300 °C, and then it dramatically decreases. The catalyst shows low catalytic activity and the NO conversion is only 77.88 % and 73.65 % at 200 °C and 550 °C, respectively. Also, the temperature window of the V_2O_5 - WO_3 /TiO₂ catalysts at 6 wt% of WO_3 loadings is narrow, which may be ascribed to that excessive WO_3 loadings trap electrons to inhibit the formation of superoxide ions (Zhang and Zhong, 2013).

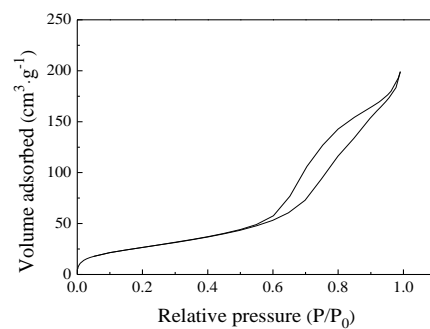


Figure 8. Nitrogen adsorption-desorption isotherms of V_2O_5 - WO_3 /TiO₂ catalyst

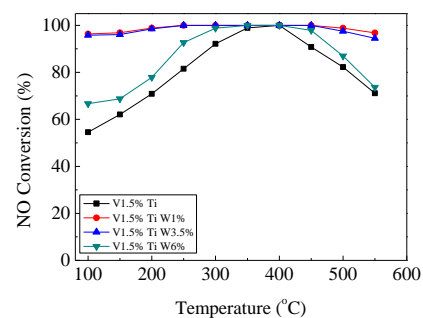


Figure 9. NO conversion for the V_2O_5 - WO_3 /TiO₂ catalysts at different WO_3 loadings

4. Conclusion

In summary, we report our works on the preparation of V_2O_5 - WO_3 / TiO_2 catalysts using a simple impregnation method and their application on the selective catalytic reduction (SCR) of NO with NH_3 . Increasing V_2O_5 loading in the V_2O_5 - WO_3 / TiO_2 catalysts can improve their catalytic activities. However, when V_2O_5 loading exceeds 2 wt%, the activity of V_2O_5 - WO_3 / TiO_2 catalyst begins to decline because high V_2O_5 loading on TiO_2 speeds up the growth of anatase- TiO_2 grains, which leads to the loss of catalytic activity. With the increase of WO_3 loadings, NO conversion is significantly promoted, however, when the WO_3 loadings reaches 6 wt%, NO conversion decreases instead due to the inhibition on the formation of superoxide ions.

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