The Effect of Cr Addition on Hg^0 Oxidation and NO Reduction over V$_2$O$_5$/TiO$_2$ Catalyst

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ABSTRACT

A series of V$_2$O$_5$-Cr$_2$O$_3$/TiO$_2$ catalysts with various chromium loadings prepared by the impregnation method were studied for Hg^0 oxidation and NO reduction. The results indicate that Cr$_2$O$_3$-modified V$_2$O$_5$/TiO$_2$ exhibited improved catalytic activity for both Hg^0 oxidation and NO reduction. In fact, with the addition of 5% Cr$_2$O$_3$ to the V/Ti catalyst, the mercury oxidation efficiency increased significantly from 6.5% to 97% in the presence of O$_2$ at 350°C, and its SCR performance also improved in the temperature range of 250°C to 400°C. To explore the effect of individual flue gas components on Hg^0 oxidation, a series of activity tests were conducted over 1% Cr-V/Ti at 350°C, and it was found that O$_2$ and HCl promote mercury oxidation, while SO$_2$ and H$_2$O exhibited a somewhat inhibitive influence. H$_2$-TPR and XPS results indicated that the enhanced catalytic performance of V$_2$O$_5$-Cr$_2$O$_3$/TiO$_2$ is attributable to the synergetic interactions between the two active phases.

Keywords: Mercury oxidation; NH$_3$-SCR; V$_2$O$_5$/TiO$_2$; Cr$_2$O$_3$.

INTRODUCTION

Mercury in flue gas discharged from coal-fired boilers has become a major environmental issue due to its volatility, persistence and bioaccumulation (Reddy *et al.*, 2012). In order to reduce mercury emissions from power plants, the U.S. Environmental Protection Agency (EPA) promulgated a federal mercury and air toxics standards (MATS) on December 21, 2011. It anticipated that stricter standards for mercury emissions would be established in the near future. The amount of Hg emissions from coal-fired power plants has thus emerged as an important issue due to the high volume of flue gas such facilities produce (Wu *et al.*, 2012). Consequently, effective and economical mercury control technologies are urgently required for coal-fired power plants.

The particle-bound mercury (Hg^p) can be effectively captured by an electrostatic precipitator (ESP) or fabric filter (FF), while oxidized mercury (Hg^{2+}) can be captured by wet flue gas desulfurization (WFGD). However, the element mercury (Hg^0) is difficult to remove using existing air pollution control devices. It was found that Hg^0 could be oxidized to Hg^{2+} to a certain extent via V$_2$O$_5$-WO$_3$(MoO$_3$)/ TiO$_2$ selective catalytic reduction (SCR) catalyst in some researchers' work, but the Hg^0 oxidation efficiency depends on the HCl concentration (Cao *et al.*, 2007; Eom *et al.*, 2008; Tsai, 2013; Stolle *et al.*, 2014). When burning subbituminous coal or lignite, the Hg^0 oxidation efficiency is relatively low due to the fairly low concentration of HCl in flue gas (Lee *et al.*, 2006; Yang and Pan, 2007). Thus, it's of great importance to enhance mercury oxidation using a V$_2$O$_5$/TiO$_2$ catalyst in flue gas with a low concentration of HCl.

Impregnating SCR catalyst surfaces with metal oxides is an effective way to enhance the mercury oxidation. However, there are only a few reported studies that have focused on the modified SCR catalyst (Zeng *et al.*, 2009; Yan *et al.*, 2011; Li *et al.*, 2017; Liu *et al.*, 2017; Yu *et al.*, 2017). Several studies have shown that chromium oxides supported on TiO$_2$ (anatase) exhibited high activity for SCR of NO with NH$_3$ in the presence of excess oxygen (Engweiler *et al.*, 1994; Schneider *et al.*, 1994; Fountzoula *et al.*, 1997; Jiang *et al.*, 2017). On the basis of this work, the catalytic performance of V$_2$O$_5$-Cr$_2$O$_3$/TiO$_2$ for NH$_3$-SCR was investigated (Fountzoula *et al.*, 1999). Besides, Cr$_2$O$_3$/TiO$_2$ was found to display excellent mercury oxidation performance in coal combustion flue gas (Kamata *et al.*, 2009). Taking these facts into consideration, it may help mercury oxidation and NO reduction when Cr$_2$O$_3$ is added to the V$_2$O$_5$/TiO$_2$ catalyst.

In this study, V$_2$O$_5$/TiO$_2$ and V$_2$O$_5$-Cr$_2$O$_3$/TiO$_2$ catalysts

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were prepared by the impregnation method. Mercury oxidation and the SCR activity of these catalysts were tested. The influence of individual flue gas components was investigated. H_2-TPR and XPS were also performed to characterize these catalysts.

METHODS

Catalyst Preparation

Catalysts were prepared by the impregnation method. $Cr(NO_3)_3 \cdot 9H_2O$ was dissolved in an aqueous solution of NH_4VO_3 in oxalic acid. To this solution, the calculated amount of titania power was added and then stirred for ten minutes. The paste obtained was dried overnight and then calcined at 500°C for 5 h in air. For these V_2O_5/TiO_2 and V_2O_5-Cr_2O_3/TiO_2 catalysts, the loading amount of V_2O_5 was 1 wt.% based on TiO_2, and the loading amount of Cr_2O_3 differed from 0.1 to 5 wt.%.

Catalyst Characterization

The temperature programmed reduction with hydrogen (H_2-TPR) was performed on a PX200 multi adsorption instrument manufactured by Tianjian Pengxiang Corporation. The H_2 signal was detected by a thermal conductivity detector (TCD). X-ray photoelectron spectra (XPS) analysis was conducted on a Thermo Escalab 250Xi instrument using Al Kα (hν = 1486.6 eV) as a radiation source at 4 KW. The binding energies (BE) were measured for Ti 2p, O 1s, V 2p, Cr 2p, and C 1s, and sample charging effects were eliminated by calibrating the observed spectra with the C 1s binding energy value of 284.8 eV.

Activity Test

The catalytic activity tests for Hg^0 oxidation were carried out in a quartz fixed-bed tube with 0.05 g catalyst samples with a particle size of 250–380 μm. The diameter of the reaction quartz tube was 6 mm. The simulated gas for these tests contained 5 vol.% of O_2, 5 ppm of HCl, 85 μg m^{-3} of Hg, 300 ppm of NO and 100 ppm of NH_3 in N_2. The gas hourly space velocity (GHSV) was 960,000 h^{-1}. The reaction temperature adopted in the mercury oxidation test was from 300 to 400°C. The concentration of mercury was determined by a portable mercury analyzer (Nippon Instruments Corporation, model EMP-2).

The catalytic activity tests for selective catalytic reduction of NO with NH_3 were performed in a fixed bed micro-reactor with a 0.1 g catalyst with a particle size of 250–380 μm. The simulated gas for these tests is consisted of 1000 ppm of NO, 5 vol.% of O_2, and 1000 ppm of NH_3 in N_2 in a GHSV of 192,000 h^{-1}. The catalytic reactions were carried out at temperatures from 250 to 450°C. The composition of the product was analyzed by a multi-component Fourier transform infrared gas analyzer (Gasmet Technologies Oy, model DX-4000).
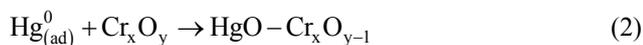
RESULTS AND DISCUSSION

Activity Test

Fig. 1 shows the catalytic activity of mercury oxidation of V/Ti and Cr-V/Ti catalysts between 300 and 400°C as the SCR catalyst usually operates in this temperature window. Mercury oxidation efficiency increases as the temperature goes up. It can be explained by the fact that more molecules are activated at higher temperatures. In some researchers' work, mercury oxidation decreases as the temperature goes up, due to the inhibition of mercury adsorption (Lee *et al.*, 2008; Straube *et al.*, 2008; Rallo *et al.*, 2012). But it was not observed for the Cr-V/Ti catalyst at reaction temperatures
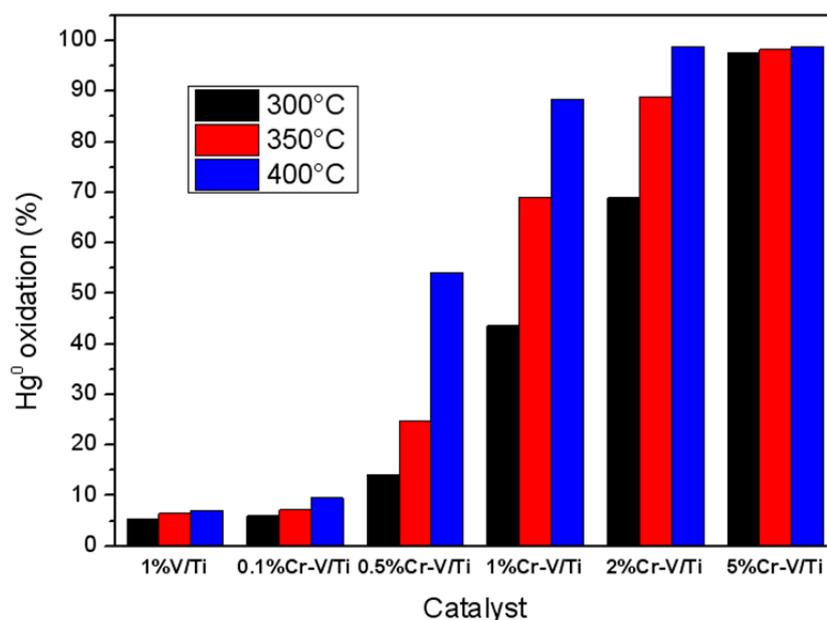
Fig. 1. The activity of mercury oxidation from 300 to 400°C over catalysts with different Cr content. Reaction condition: $[Hg^0] \approx 85$ μg m^{-3}, $[O_2]$ = 5 vol.%, [HCl] = 5 ppm, [NO] = 300 ppm, $[NH_3]$ = 100 ppm, balanced gas = N_2, GHSV = 960000 h^{-1}.

from 300 to 400°C. More important, mercury oxidation is greatly affected by the Cr content. The mercury oxidation efficiency increases from 6.5% to 69.0% at 350°C with the addition of Cr_2O_3 to a 1% V/Ti catalyst. For 5% Cr-V/Ti, Hg^0 oxidation exceeds 97% at testing temperatures. XRD results indicated that chromium oxides were highly dispersed on the catalyst surface even for 5% Cr loading. The super mercury oxidation activity should be attributed to the excellent oxidation ability of chromium oxides. The Mars-Maessen mechanism may be used to explain the mercury oxidation in this reaction atmosphere (Granite *et al.*, 2000; Liu *et al.*, 2011). Gas-phase element mercury is firstly adsorbed on the chromium oxide of the catalyst surface and then reacts with lattice oxygen to form weakly adsorbed mercuric oxide. The consumed lattice oxygen is refilled with the gas-phase oxygen, as shown in Eq. (1) to Eq. (3).

$$Hg^0_{(g)} \leftrightarrow Hg^0_{(ad)} \tag{1}$$

$$Hg^0_{(ad)} + Cr_xO_y \rightarrow HgO - Cr_xO_{y-1} \tag{2}$$

$$2HgO - Cr_xO_{y-1} + O_2 \rightarrow 2HgO_{(g)} \uparrow + 2Cr_xO_y \tag{3}$$

The effect of the Cr addition on NO conversion is illustrated in Fig. 2. It can be seen that the Cr addition to the V/Ti catalyst is beneficial to the NO reduction at low-temperatures. The NO conversion at 300°C of 1% V/Ti is 24.0%, and it increases with the Cr loading. For 5% Cr-V/Ti, the NO conversion reaches 86.4%. Compared to the NO conversion of 43.9% at 350°C for 1% V/Ti, the NO conversion increases to 77.5% for 0.5% Cr-V/Ti and 92.7% for 1% Cr-V/Ti. The enhanced catalytic activity should be attributed to the excellent oxidation ability of chromium oxides. However, the NO conversion decreased with a further

increase in the Cr loading. The NO conversion of 1% Cr-V/Ti at 400°C is 90.4%, but it decreased to 84.2% and 81.7% for 2% Cr-V/Ti and 5% Cr-V/Ti respectively. The catalytic activity is even lower than for the V/Ti catalyst when the Cr loading is above 1% at 450°C. Similar phenomenon was observed in Fountzoula's work (Fountzoula *et al.*, 1999). In these situations, the direct oxidation of NH_3 by O_2 and the production of N_2O may take place due to the strong redox properties of chromium oxides. When primarily considering the activity in the typical temperature window for SCR systems in power plants (350–370°C), 1% Cr-V/Ti shows the best catalytic performance.

Effect of Individual Flue Gas Components

To explore the effect of individual flue gas components on Hg^0 oxidation, a series of activity tests were conducted over 1% Cr-V/Ti at 350°C. As can be seen in Fig. 3, in a pure N_2 atmosphere, the mercury oxidation with 1% Cr-V/Ti is negligible. When O_2 is added to the reaction atmosphere, mercury oxidation is significantly improved at 350°C. Mercury oxidation increases to 40% in 2% O_2 and 61.2% in 5% O_2, which may be explained by the aforementioned Mars-Maessen mechanism. HCl is thought to be the most important flue gas component to affect mercury oxidation since the main oxidized mercury species in flue gas would be $HgCl_2$ (Cao *et al.*, 2007). HCl reacts with weakly adsorbed mercuric oxide to form volatile mercuric chloride, and mercury chloride then desorbs from the catalyst surface (Liu *et al.*, 2011). The addition of 5 ppm of HCl and 10 ppm of HCl increases the Hg oxidation efficiency to 43.5% and 56.5%, respectively. When 10 ppm of HCl and 5% O_2 were simultaneously added in N_2, the mercury oxidation efficiency increased to 87.1%. Considering the high gas hourly space velocity, it can be seen that the 1% Cr-V/Ti catalyst has an excellent mercury oxidation ability even in a low HCl concentration atmosphere. In the absence of O_2,
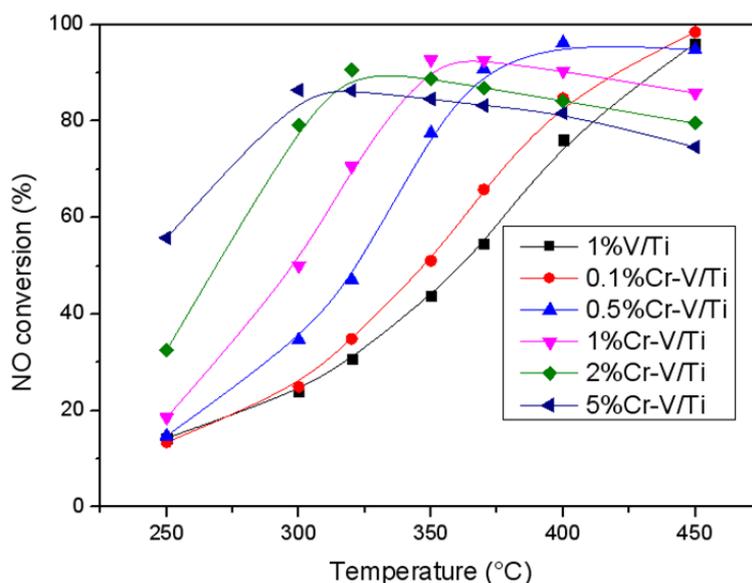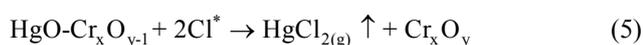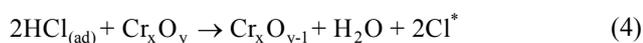

Fig. 2. The activity of NO conversion as a function of temperature over catalysts with different Cr content. Reaction condition: [NO] = [NH₃] = 1000 ppm, [O₂] = 5 vol.%, balanced gas = N₂, GHSV = 192000 h⁻¹.
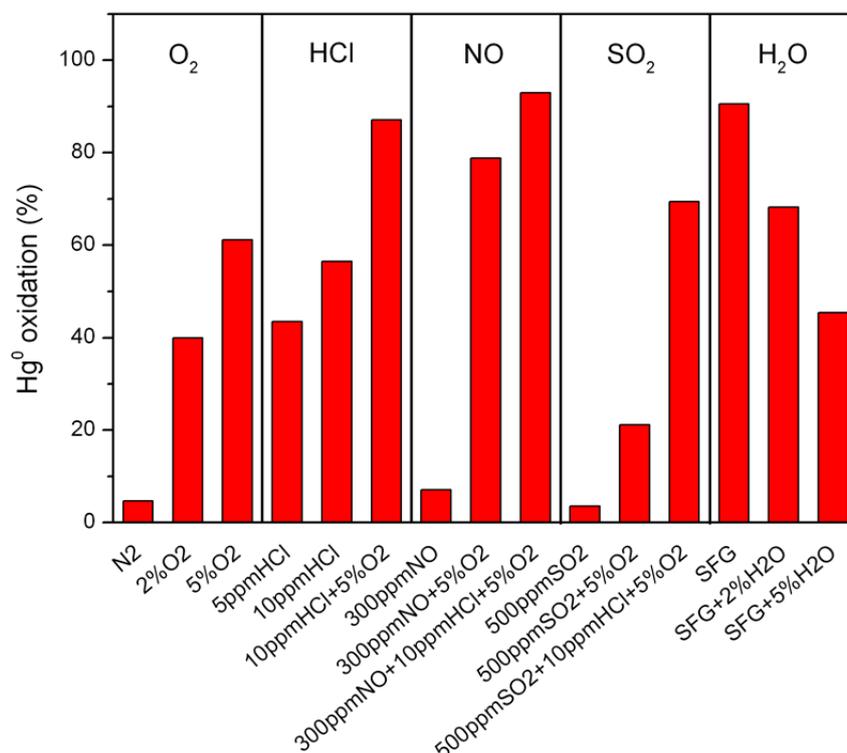
Fig. 3. Effect of individual flue gas components on Hg^0 oxidation over 1% Cr-V/Ti at 350°C.

HCl was firstly adsorbed on the catalyst surface, then reacted with lattice oxygen to form active chlorine species, and finally reacted with Hg^0 to complete the mercury oxidation. In the presence of O_2, the promoting effect of HCl on mercury oxidation was more obvious: The Hg oxidation efficiencies were all above 90% with the addition of 1 ppm of HCl, 5 ppm of HCl and 10 ppm of HCl in 5% O_2, which exhibited an effective Hg^0 oxidation even with the low chlorine concentration. The Hg^0 oxidation efficiency in the concurrent presence of both 1 ppm of HCl and 5% O_2 was greater than the sum of the individual Hg^0 oxidation efficiency with either 1 ppm HCl or 5% O_2 alone. It therefore suggested a synergetic promotion between HCl and O_2, and the mechanism for mercury oxidation might also have changed.

More specifically, with the presence of HCl and O_2, reactive intermediates of Cl_2 or Cl may form due to the catalytic effect of chromium oxide to Deacon reaction, as shown in Eq. (4), and these species have an excellent promotional effect on mercury oxidation. Besides, with the presence of HCl , the weakly adsorbed HgO reacts with active chlorine to form volatile mercuric chloride, and mercury chloride then desorbs from the catalyst surface, as shown in Eq. (5), promoting the reaction between O_2 and Hg^0. In addition, the active chlorine may directly react with gaseous Hg^0 to form $HgCl_2$, as shown in Eq. (6).

$$2HCl_{(ad)} + Cr_xO_y \rightarrow Cr_xO_{y-1} + H_2O + 2Cl^* \tag{4}$$

$$HgO\text{-}Cr_xO_{y-1} + 2Cl^* \rightarrow HgCl_{2(g)} \uparrow + Cr_xO_y \tag{5}$$

$$Hg^0_{(g)} + 2Cl^* \rightarrow HgCl_{2(g)} \tag{6}$$

NO was reported to promote Hg^0 oxidation in some researchers' work (Li *et al.*, 2011; Fan *et al.*, 2012), as it can be oxidized and form species such as NO^+ and NO_2 on some catalysts (Hu *et al.*, 2017). For the Cr-V/Ti catalyst, introducing NO into pure N_2 does not create an obvious influence on Hg^0 oxidation. However, a promotional effect is observed when NO is added in the presence of O_2. Mercury oxidation increases to 78.8% from 61.2% when 300 ppm of NO is added. The promotional effect is also observed when O_2 and HCl simultaneously exist in the reaction atmosphere. Mercury oxidation increases to 92.9% from 87.1% when 300 ppm of NO is added.

The influence of SO_2 on mercury oxidation with different catalysts is quite complex. Some researchers observed a promotional effect (Fan *et al.*, 2010, 2012), while other researchers observed an inhibitory (Cao *et al.*, 2007; Zhuang *et al.*, 2007; Wen *et al.*, 2011) or negligible effect (Li *et al.*, 2008). In our experiment, because mercury oxidation is negligible in pure N_2, no obvious effect was observed when SO_2 was introduced. However, SO_2 has a significant inhibitory effect on Hg^0 oxidation in the gas flow containing O_2 or O_2 and HCl. When 500 ppm of SO_2 is added to 5% O_2, mercury oxidation decreases to 21.2%. This remarkable inhibitive effect is also found in 10 ppm of HCl and 5% O_2, and mercury oxidation decreases to 69.4% when 500 ppm of SO_2 is added. The inhibitory effect of SO_2 is probably due to strong competitive adsorption with Hg^0 (Zhuang *et al.*, 2007).

Finally, the mercury oxidation in simulated dry and simulated wet flue gas was tested. The simulated flue gas consists of 5 vol.% O_2, 10 ppm of HCl, 300 ppm of NO, and 500 ppm of SO_2. In dry SFG, mercury oxidation is 90.6%. However, it decreases to 68.2% and 45.5% when 2% or 5% H_2O is added to SFG, which indicates that H_2O also strongly suppresses mercury oxidation with aover Cr-V/Ti catalyst. The inhibitive effect of H_2O on mercury oxidation was also found in other researchers' work (Li *et al.*, 2012; Zhang *et al.*, 2014). Competitive adsorption to the catalyst surface may exist between H_2O and Hg^0. It should be noted that in these tests, the gas hourly space velocity (GHSV) was as high as 960000 h^{-1} in order to distinctly illustrate the effects of different flue gas components. GHSV is about 10000 h^{-1} in coal-fired power plants, and mercury oxidation should be much higher.

Catalyst Characterization

Temperature programmed reduction with hydrogen (H_2-TPR) was performed to examine the interactions between two active phases for the Cr-V/Ti catalyst. As can be seen in Fig. 4, the 1% V/Ti catalyst only possesses one peak at 455°C. Two reduction peaks are detected at 356 and 444°C for 1% Cr/Ti. The low temperature peak should be due to the reduction of Cr^{6+} to Cr^{5+}, while the high temperature peak should be due to the reduction of Cr^{5+} to Cr^{3+}. In other researchers' work (Fountzoula *et al.*, 1997), two reduction peaks were also found for the Cr/Ti catalyst with similar Cr loadings. As for 1% Cr-V/Ti, only one reduction peak at 426°C was detected, indicating that there is a cooperative effect between V and Cr. The shift of the main reduction peak at a low temperature indicates that the redox ability is improved after adding Cr to the V/Ti catalyst, which contributes to better catalytic activity of the Cr-V/Ti catalyst. It can be verified by XPS analysis, as illustrated in Fig 5. A similar phenomenon was found for the 2% Cr/Ti and 2% Cr-V/Ti catalysts. 2% Cr/Ti possessed two reduction peaks at 325 and 419°C, respectively, and 2% Cr-V/Ti possessed one reduction peak at 415°C.

X-ray photoelectron spectra (XPS) analysis was conducted to elucidate the surface nature of the active sites over the catalysts. As shown in Fig. 5(a), the XPS spectra of the V 2p exhibit obvious transformations. According to other researchers' work (Dupin *et al.*, 2000; He *et al.*, 2009), the XPS curve of V 2p can be fitted into two peaks, for which the peak at the binding energy of ≈515.9 eV is attributable to the photoemission of V^{4+} and the peak at the binding energy of ≈517.1 eV is attributable to V^{5+}. For 1% V/Ti, the ratios of V^{5+} and V^{4+} were 43.0 and 57.0% respectively. When Cr is added to the V/Ti catalyst, the ratio of V^{5+} significantly increases. The ratio of V^{5+} increases to 52.4 and 71.3% for 1% Cr-V/Ti and 2% Cr-V/Ti. It can be inferred that a certain amount of V^{4+} is oxidized to V^{5+} due to the interaction between Cr and V. And Cr-O-V interaction species may be formed between the isolated Cr^{6+} species and the well-dispersed V^{5+} species (Fountzoula *et al.*, 1999).

Fig. 5(b) shows the XPS spectrums of Cr 2p. According to other researchers' work (Fountzoula *et al.*, 1999), for Cr-V/Ti catalysts, chromium existed in at least two valence states, namely, Cr^{6+} and Cr^{3+}. The peak at the blinding energy of ≈576.9 eV represents Cr^{3+}, and the peak at blinding energy of ≈579 eV represents Cr^{6+}. And some amount of Cr^{5+} (577.5–578.5 eV) may also exist. Compared to 1% Cr/Ti, the intensity of the peak representing Cr^{6+} showed a slight trend of decreasing, which is in accordance with the increase in V^{5+}.

CONCLUSIONS

The V_2O_5-Cr_2O_3/TiO_2 catalysts showed both higher Hg^0 oxidation and NO conversion than the V_2O_5/TiO_2 catalysts. The effects of individual flue gas components on Hg^0 oxidation were studied. It was found that O_2 and HCl promote mercury oxidation, and NO also had a positive effect. SO_2 could inhibit mercury oxidation, and the inhibitive effect was probably due to the competitive adsorption with Hg^0. H_2O was also found to inhibit mercury oxidation. H_2-TPR results showed a shift in the reduction peak at a low
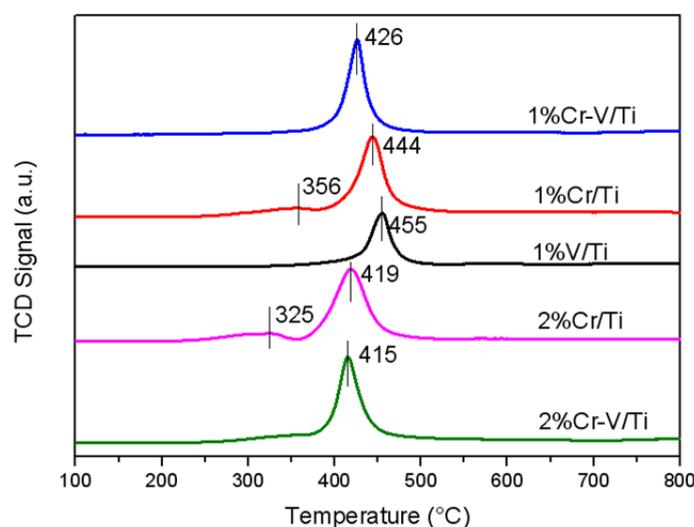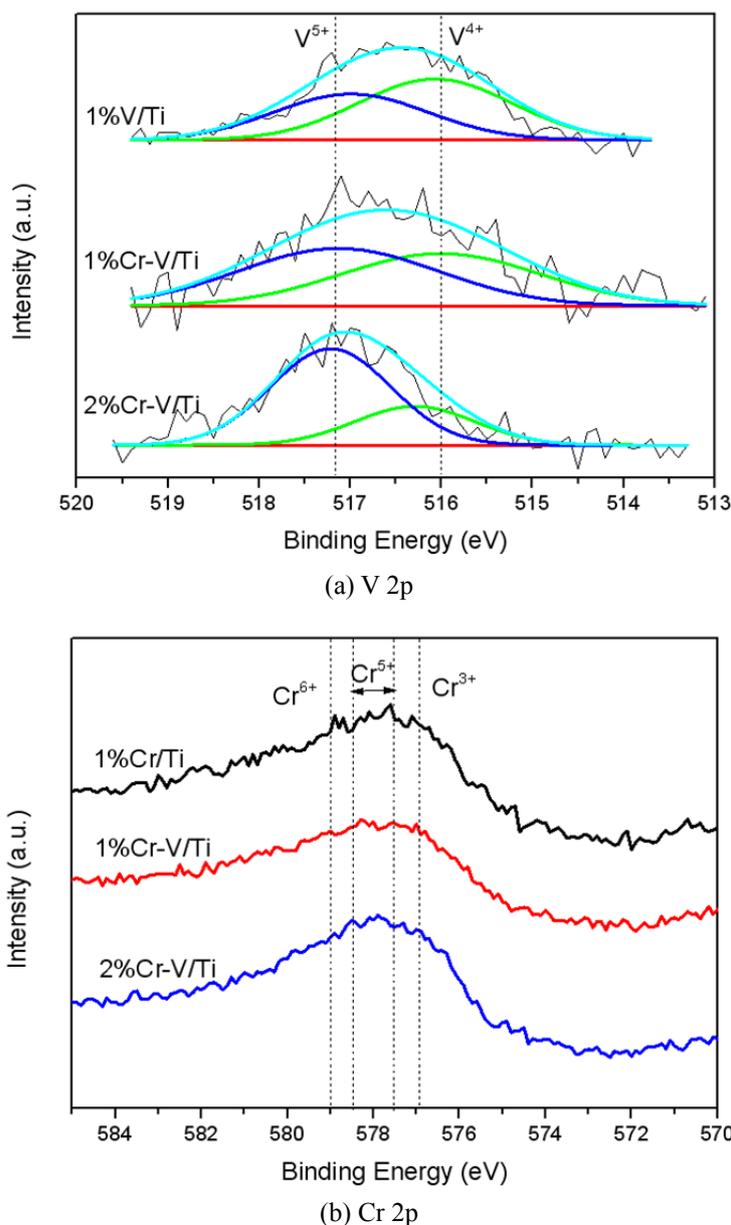

Fig. 4. H_2-TPR profiles of V/Ti and Cr-V/Ti catalysts.

(a) V 2p

(b) Cr 2p

Fig. 5. XPS spectra of V 2p (a) and Cr 2p (b) of V/Ti and Cr-V/Ti catalysts.

temperature for the Cr-V/Ti catalyst. XPS results also indicated synergy between the two active phases.

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