

# Effect of V<sub>2</sub>O<sub>5</sub> Supported on the Sulfated TiO<sub>2</sub> for Low-temperature SCR of NO by NH<sub>3</sub>

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## Abstract

V<sub>2</sub>O<sub>5</sub>-WO<sub>3</sub>/TiO<sub>2</sub>-SO<sub>4</sub><sup>2-</sup> catalysts were prepared by wet impregnation method with sulfated nano-TiO<sub>2</sub>. Different acid amounts, activities, sulfur tolerance and regeneration of the catalysts were tested. It was found that the low-temperature catalytic activity of V<sub>2</sub>O<sub>5</sub>-WO<sub>3</sub>/TiO<sub>2</sub>-SO<sub>4</sub><sup>2-</sup> was improved after modified with 0.4M H<sub>2</sub>SO<sub>4</sub>. BET and XRD results showed that the specific surface area of catalysts decreased after sulfating without crystal form changing. The catalytic activity of V<sub>2</sub>O<sub>5</sub>-WO<sub>3</sub>/TiO<sub>2</sub>-SO<sub>4</sub><sup>2-</sup> catalyst was better than the original, which decreased from 95% to 90% in 50h under the condition of 300ppm SO<sub>2</sub> at temperature of 180°C. FT-IR and TG results showed that the deactivation by SO<sub>2</sub> was due to the ammonium sulfate forming during the SCR reaction. The activity of poisoned catalysts could be recovered to the fresh level after calcined at 490°C.

**Keywords:** Acidification; SCR; Sulfur Tolerance; Regeneration

## 1 INTRODUCTION

Nitrogen oxide (NO<sub>x</sub>) emissions are the main sources of air pollution which have harmful effects on the environment and human health (Mauzerall D L, et al., 2005). Selective catalytic reduction (SCR) of nitrogen oxides with ammonia is one of the widely used processes for cleaning the flue gas from stationary sources and diesel engines (Teng H, 1999). The general SCR reaction is:  $4\text{NO} + 4\text{NH}_3 + \text{O}_2 \rightarrow 4\text{N}_2 + 6\text{H}_2\text{O}$ .

SCR catalysts are the most critical factor in the selective catalytic reduction process. A large number of SCR catalysts have been studied in the literature that V<sub>2</sub>O<sub>5</sub>/TiO<sub>2</sub> catalyst has been widely applied in NO<sub>x</sub> removal. Such commercial catalyst has excellent activity in the SCR reaction within a narrow high temperature range (Busca G, et al., 1998). However, it is necessary to locate the SCR unit upstream of the desulfurizer and electrostatic precipitator in order to avoid reheating of the flue gas as well as deposition of dust on the catalyst (H. Bosch, 1988). For this reason, it is urgent to develop a superior SCR catalyst that exhibits high activity at wide low temperature between 120-400°C to ensure the catalyst deactivation could be avoided. Previous studies have shown that the SCR activity could be improved by the catalysts doping with SO<sub>4</sub><sup>2-</sup> ion as TiO<sub>2</sub> promoter. The support TiO<sub>2</sub> impregnated with H<sub>2</sub>SO<sub>4</sub> has high activity in high temperature reactions between 400°C and 600°C (Chen J P, 1993; Jung S M, 2000). Alemany L J et al (Alemany L J, 1996) found that the specific surface area and total pore volume of TiO<sub>2</sub> was increased after doping the SO<sub>4</sub><sup>2-</sup>. Moreover, It has been reported that Brønsted acid which provided by SO<sub>4</sub><sup>2-</sup> on TiO<sub>2</sub> could maintained a superior SCR activity at low temperature (Sun Q, 2008; Ciambelli P, 1996). It has been also proposed that SO<sub>4</sub><sup>2-</sup> produces a strong acidic site which could lead to the high reactivity in the SCR reaction (Y. Tsutomu, 1990).

According to many reports in the literature, it has been shown that SO<sub>4</sub><sup>2-</sup> could increase the acidity and reactivity of SCR catalysts. However, until now, no studies about sulfating V<sub>2</sub>O<sub>5</sub>-WO<sub>3</sub>/TiO<sub>2</sub> directly have been proposed. In this article, as a support material, TiO<sub>2</sub> anatase was modified with SO<sub>4</sub><sup>2-</sup> directly. V<sub>2</sub>O<sub>5</sub>-WO<sub>3</sub>/TiO<sub>2</sub>-SO<sub>4</sub><sup>2-</sup> catalysts were carried out by two-step wet impregnation method. Different acid amounts, activity, sulfur tolerance and regeneration of the catalysts were tested. We further investigated the mechanism of SO<sub>2</sub> deactivation process of V<sub>2</sub>O<sub>5</sub>-WO<sub>3</sub>/TiO<sub>2</sub>-SO<sub>4</sub><sup>2-</sup> catalysts using BET, FT-IR and TG.

## 2 EXPERIMENT

### 2.1 Catalyst Preparation

The  $V_2O_5$ - $WO_3$ / $TiO_2$ - $SO_4^{2-}$  catalysts were prepared by two-step wet impregnation method. To obtain the sulfated catalyst, required amounts of  $TiO_2$  were added to a 200mL beaker within 50mL  $H_2SO_4$  solution (0.0-1.0M). The support solution was stirred continuously for 2h. The resulting solids were oven dried at 105°C for 4h, then calcined at 490°C for 3h. The sulfated supports were ground and sieved to 100 mesh size.

$V_2O_5$ - $WO_3$ / $TiO_2$ - $SO_4^{2-}$  catalysts employed in the present study containing  $V_2O_5$  and  $WO_3$  were prepared by aqueous solution of  $NH_4VO_3$  and  $(NH_4)_{10}W_{12}O_{41}$  diluted in oxalic acid in a desired proportion. The calculated amount of sulfated  $TiO_2$  support was impregnated in this solution by stirring for 2h. The resulting materials were oven dried at 105°C for 4h, then calcined at 490°C for 3h. The catalysts were ground and sieved (20-40 mesh) to obtain homogeneous powder.

### 2.2 Catalyst Characterization

The surface areas of the catalyst powders were measured by physical nitrogen adsorption at -196°C using Micromeritics Gemini V. The catalysts (0.1 – 0.2 g) were degassed at 110°C for 1 h.

The X-ray diffraction (XRD) measurements were performed with Cu Ka ( $k = 0.15406$  nm) radiation. The catalysts were run at 2 $\theta$ , ranging from 10° to 80° with a step size of 0.020° and a time step of 1.0 s using Bruker D8.

FT-IR spectra were recorded using a Bruker VECTOR22 spectrometer with a wave number range from 400 to 4000  $cm^{-1}$  and a spectral resolution of 4  $cm^{-1}$ . The samples were pressed with KBr.

Thermo gravimetric analysis (TG) for the different series of catalyst was carried out in a TG/DTA6300 with sensitivity of 0.1  $\mu g$ . The samples were tested under air atmosphere from 25 to 1000°C with heating rate of 10°C/min<sup>-1</sup>.

### 2.3 Catalytic Activity Test

The SCR activity of the catalysts for NO removal with  $NH_3$  was carried out in a fixed-bed flow reactor (i.d. 6 mm). A temperature controller and thermo couple which used to control the temperature of the reactor were inserted directly into the catalyst bed. The reactant gas composition was as follows: 700ppm NO, 700ppm  $NH_3$ , 300ppm  $SO_2$ , 5vol%  $O_2$  and  $N_2$  as balance. The gas hourly space velocity (GHSV) was 27000h<sup>-1</sup>. The feed gases were mixed before entering the reactor. The reactants and products were analysed on line using a Nitrogen Oxides Analyser, 42i-HL (Thermo Electron Corporation).

## 3 RESULTS AND DISCUSSION

### 3.1 Acid Amounts on $V_2O_5$ - $WO_3$ / $TiO_2$ - $SO_4^{2-}$ Catalysts

The catalysts modified with different acid amounts were prepared to compare the activities. The reaction conditions were as follows: 700ppm NO, 700ppm  $NH_3$ , 5%  $O_2$ ,  $N_2$  balance, GHSV of 27000h<sup>-1</sup>. According to the results from Fig.1, the activity of  $V_2O_5$ - $WO_3$ / $TiO_2$  catalyst was obtained 30.7%, 60.5% and 90% at low temperature of 140,160 and 180°C. However, NOx removal activity of catalyst supported on sulfated  $TiO_2$  (0.4M  $H_2SO_4$ ) has been enhanced even up to 41%, 70% and 95% at low temperature of 140,160 and 180°C. It also indicated that  $SO_4^{2-}$  doping were favorable to improve the low-temperature activities of  $V_2O_5$ - $WO_3$ / $TiO_2$ - $SO_4^{2-}$  catalysts. However, further increase of the amount of acid to 0.5mol/L lowered the activity of the catalysts. It may be due to the presence of excess sulfate species which cover the active sites on the catalyst surface.

Therefore, it could be concluded that sulfuric acid doping had positive effect on the low-temperature activity of the catalysts (Choo S T, 2000). It was clearly observed that sulfuric species played a certain role for the Brønsted acid sites on the surface of catalysts. As for SCR reaction process,  $NH_3$  adsorption was enhanced by the increase of the amounts and strength of the sulfate acid sites on the catalyst surface. Moreover, the effect of catalyst surface acidity was favorable for NO removal activity.

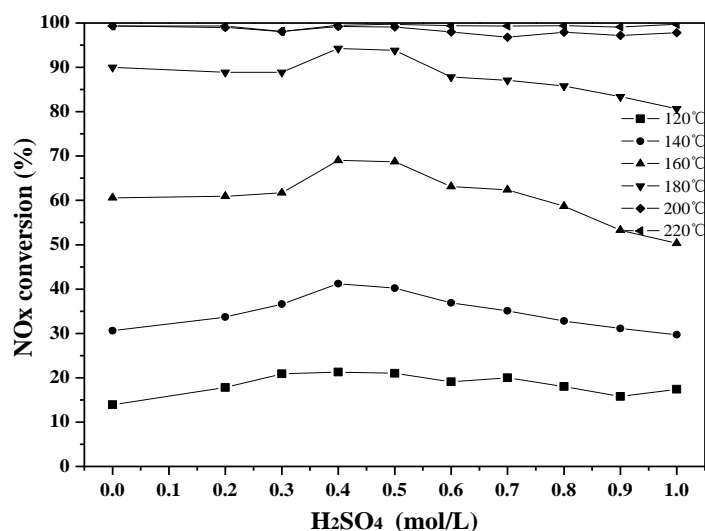


FIG. 1 NO CONVERSION OVER  $V_2O_5$ - $WO_3$ /TiO<sub>2</sub>- $SO_4^{2-}$  CATALYSTS

### 3.2 $SO_2$ Tolerance and Regeneration of $V_2O_5$ - $WO_3$ /TiO<sub>2</sub> ( $-SO_4^{2-}$ ) Catalysts

The effect of 300ppm  $SO_2$  on SCR activity of  $V_2O_5$ - $WO_3$ /TiO<sub>2</sub> and  $V_2O_5$ - $WO_3$ /TiO<sub>2</sub>- $SO_4^{2-}$  at temperature of 180°C is illustrated in Fig.2. It could be seen that the steady NOx conversion of  $V_2O_5$ - $WO_3$ /TiO<sub>2</sub> and  $V_2O_5$ - $WO_3$ /TiO<sub>2</sub>- $SO_4^{2-}$  were obtained 90% and 95% under the condition without  $SO_2$ , respectively. However, in the presence of  $SO_2$ , the catalytic activity of  $V_2O_5$ - $WO_3$ /TiO<sub>2</sub>- $SO_4^{2-}$  was improved slightly to 97.7%. The promotion behavior of catalyst activity may be due to the increase of the sulfuric acid sites that enhanced the  $NH_3$  adsorption (Motomobu Kobayashi, 2006). It could be observed that the NOx removal activity of the  $V_2O_5$ - $WO_3$ /TiO<sub>2</sub> decreased rapidly than that of  $V_2O_5$ - $WO_3$ /TiO<sub>2</sub>- $SO_4^{2-}$ . In the case of 300ppm  $SO_2$ , approximately 76% and 90% of NO conversion by  $V_2O_5$ - $WO_3$ /TiO<sub>2</sub> and  $V_2O_5$ - $WO_3$ /TiO<sub>2</sub>- $SO_4^{2-}$  remained after 50h. It was generally considered that  $SO_2$  deactivation mechanism of SCR catalysts was mainly due to two aspects (Giakoumelou I, 2006). (1) Metal sulfate formed by  $SO_2$  and metal oxide (active phase or support) lead to catalyst chemical deactivation. (2) Deposition on catalyst surface of ammonium sulfate/bisulfate lead to physical deactivation caused by  $SO_2$  and  $NH_3$  at low temperature.

To investigate the deactivation mechanism of  $SO_2$  on the catalyst activity, the used catalyst was then regenerated by calcined at 490°C for 3h with a continuous flow of  $N_2$ . As shown in Fig.3, the NO conversion of  $V_2O_5$ - $WO_3$ /TiO<sub>2</sub>- $SO_4^{2-}$  could be approximately recovered to the original levels during the temperature of 140-200°C after thermo regeneration.

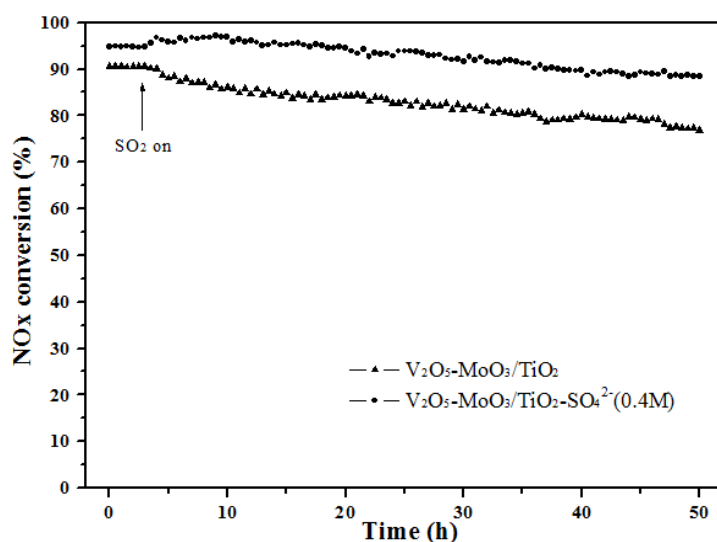


FIG. 2 EFFECT OF  $SO_2$  ON NO CONVERSION OVER  $V_2O_5$ - $WO_3$ /TiO<sub>2</sub> ( $-SO_4^{2-}$ ) CATALYSTS

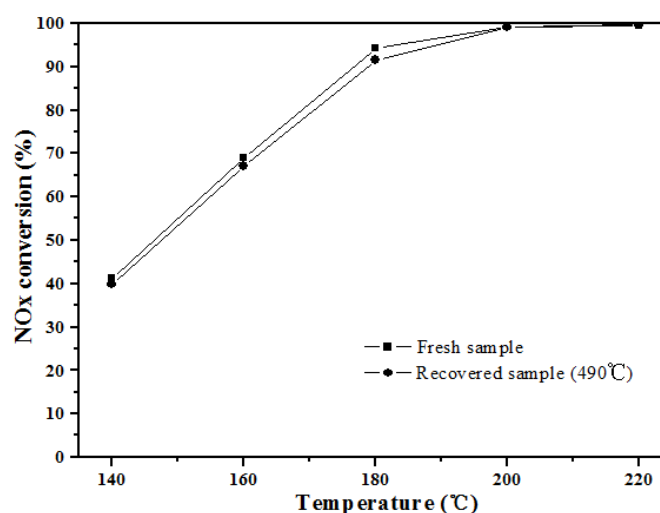


FIG. 3 ACTIVITY TEST ON RECOVERED  $V_2O_5-WO_3/TiO_2-SO_4^{2-}$  CATALYSTS

### 3.3 BET Surface Area and X-ray Diffraction Measurements

BET surface areas of each catalyst are summarized in Table 1. For  $V_2O_5-WO_3/TiO_2$  catalyst, the specific surface area was observed to be  $85 \text{ m}^2/\text{g}$ . After modified with  $0.2\text{M } H_2SO_4$ , the specific surface area of  $V_2O_5-WO_3/TiO_2-SO_4^{2-}$  catalysts decreased rapidly to  $66 \text{ m}^2/\text{g}$ . With the increasing concentration of sulfuric acid, the specific surface area of catalysts decreased slightly. It may be caused by nano agglomerated particles on  $TiO_2$  that plugged part of the active sites on catalyst surface. However, as the mass fraction of  $SO_4^{2-}$  increased, the SCR activity of catalysts did not enhance further with the addition of  $SO_4^{2-}$ .  $V_2O_5-WO_3/TiO_2$  modified with  $0.4\text{mol/L } H_2SO_4$  amongst all the catalysts showed the best SCR activity.

TABLE 1 BET SURFACE AREA OF CATALYSTS

| Mum. | Sample                                     | BET ( $\text{m}^2 \text{g}^{-1}$ ) |
|------|--|------------------------------------|
| 1    | $V_2O_5-WO_3/TiO_2$                        | 85                                 |
| 2    | $V_2O_5-WO_3/TiO_2-SO_4^{2-}(0.2\text{M})$ | 66                                 |
| 3    | $V_2O_5-WO_3/TiO_2-SO_4^{2-}(0.3\text{M})$ | 65                                 |
| 4    | $V_2O_5-WO_3/TiO_2-SO_4^{2-}(0.4\text{M})$ | 61                                 |
| 5    | $V_2O_5-WO_3/TiO_2-SO_4^{2-}(0.5\text{M})$ | 61                                 |
| 6    | $V_2O_5-WO_3/TiO_2-SO_4^{2-}(0.6\text{M})$ | 62                                 |
| 7    | $V_2O_5-WO_3/TiO_2-SO_4^{2-}(0.7\text{M})$ | 58                                 |
| 8    | $V_2O_5-WO_3/TiO_2-SO_4^{2-}(0.8\text{M})$ | 55                                 |
| 9    | $V_2O_5-WO_3/TiO_2-SO_4^{2-}(0.9\text{M})$ | 51                                 |
| 10   | $V_2O_5-WO_3/TiO_2-SO_4^{2-}(1.0\text{M})$ | 48                                 |

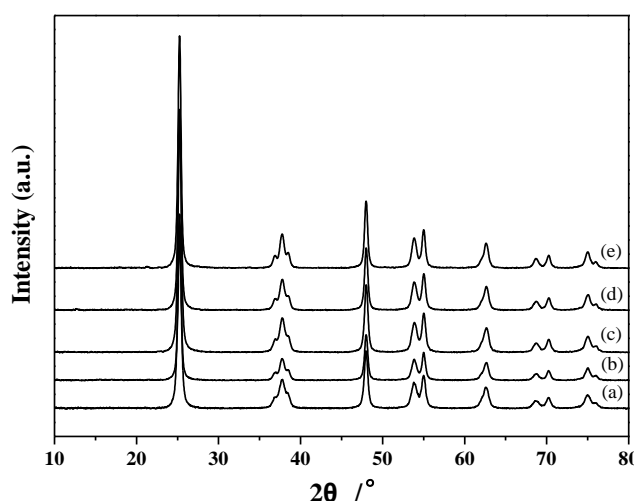


FIG. 4 X-RAY DIFFRACTION PATTERNS OF THE  $V_2O_5-WO_3/TiO_2$ (a)  $V_2O_5-WO_3/TiO_2-SO_4^{2-}(0.2\text{M})$ (b),  $V_2O_5-WO_3/TiO_2-SO_4^{2-}(0.4\text{M})$ (c),  $V_2O_5-WO_3/TiO_2-SO_4^{2-}(0.7\text{M})$ (d),  $V_2O_5-WO_3/TiO_2-SO_4^{2-}(1.0\text{M})$ (e) CATALYST

The XRD patterns of  $V_2O_5-WO_3/TiO_2$  with  $SO_4^{2-}$  loadings are shown in Fig.4. For all the catalyst samples, only anatase  $TiO_2$  oxide was detected with the increasing amounts of the sulfuric acid. It was indicated that sulfuric catalysts remained its amorphous structure with no other distinctively obvious diffraction peaks, which suggested that  $V_2O_5$  was highly dispersed on the catalyst surface.

### 3.4 BET Surface Area and Pore Structure Measurements of Poisoned Catalysts

BET surface area and pore structure measurements of poisoned catalysts are listed in Table 2. The surface area of the catalyst poisoned by  $SO_2$  was confirmed to be lowered than that of the fresh catalyst. For poisoned catalyst, the specific surface area, pore volume and average pore diameter were decreased to  $57 \text{ m}^2/\text{g}$ ,  $0.23 \text{ cm}^3/\text{g}$  and  $13.7 \text{ nm}$ , respectively. It appeared that poisoning substances generated by  $SO_2$  would cover the active sites on the catalyst surface, which reduced the physical properties and SCR activity of the catalyst. However, after calcined at  $250^\circ\text{C}$  and  $490^\circ\text{C}$ , the specific surface area and average pore diameter increased to  $63 \text{ m}^2/\text{g}$ ,  $14.7 \text{ nm}$  and  $14.8 \text{ nm}$ , respectively. It was clearly indicated that poisoning substances could be decomposed at the temperature of  $250^\circ\text{C}$  and  $490^\circ\text{C}$ , which improved all the physical properties to the original levels.

TABLE 2 BET SURFACE AREA AND PORE STRUCTURE OF POISONED CATALYSTS

| Mum. | Sample  | BET ( $\text{m}^2/\text{g}$ ) | Pore volume ( $\text{cm}^3/\text{g}$ ) | Average pore diameter (nm) |
|------|---|-------------------------------|--|----------------------------|
| 1    | $V_2O_5-WO_3/TiO_2-SO_4^{2-}(0.4\text{M})$                                | 61                            | 0.25                                   | 14.8                       |
| 2    | $V_2O_5-WO_3/TiO_2-SO_4^{2-}(0.4\text{M})$ -Poisoned                      | 57                            | 0.23                                   | 13.7                       |
| 3    | $V_2O_5-WO_3/TiO_2-SO_4^{2-}(0.4\text{M})$ -Poisoned- $250^\circ\text{C}$ | 63                            | 0.26                                   | 14.7                       |
| 4    | $V_2O_5-WO_3/TiO_2-SO_4^{2-}(0.4\text{M})$ -Poisoned- $490^\circ\text{C}$ | 63                            | 0.27                                   | 14.8                       |

### 3.5 FT-IR and TG Analyses of Poisoned Catalysts

To identify the deactivation mechanism of  $V_2O_5-WO_3/TiO_2-SO_4^{2-}$  catalysts poisoned by  $SO_2$  for the SCR of NO with  $NH_3$ , FT-IR spectra of fresh and spent catalysts are shown in Fig.5. All the catalysts exhibited vibration absorptions at wave numbers of  $1635 \text{ cm}^{-1}$ , which could be attributed to the absorbed water in the catalysts and KBr (Shen, 2009). Compare to the fresh catalyst, a new band at  $1398 \text{ cm}^{-1}$  was appeared for poisoned catalyst, which could be assigned to  $NH_4^+$  species chemisorbed on the Brønsted acid sites (Sherrie Elzey, et al., 2008; Zhu, et al., 2001). Simultaneously, two new bands at  $1137 \text{ cm}^{-1}$  and  $1046 \text{ cm}^{-1}$  which were classified to the characteristic frequencies of  $SO_4^{2-}$  ion appeared (Xie, 2003; Daniela Pietrogiaconi, et al., 2002). Therefore, the results above demonstrated that ammonium sulfate species would be formed on the catalysts during the SCR reaction in the presence of  $SO_2$ , which covered and clogged the catalyst sites and pores. Moreover, for recovered at  $250^\circ\text{C}$  catalyst, the intensity of the bands at  $1398 \text{ cm}^{-1}$ ,  $1137 \text{ cm}^{-1}$  and  $1046 \text{ cm}^{-1}$  were further weakened, which suggested that the amount of the  $NH_4^+$  and  $SO_4^{2-}$  ions decreased with temperature increasing. In addition, there were no sulfate species bands detected on catalyst recovered at  $490^\circ\text{C}$ , which means that the deactivating ammonium sulfate salt agents could be decomposed at  $490^\circ\text{C}$  and above. It also demonstrated that there were no any metal sulfates formed by  $SO_2$  at low temperature in the SCR reaction.

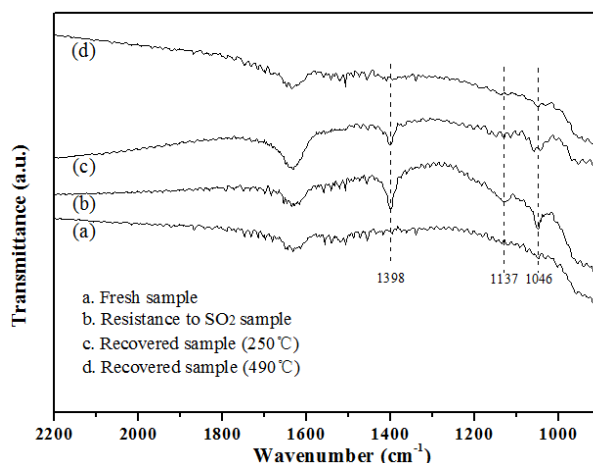


FIG. 5 FT-IR SPECTRA OF POISONED  $V_2O_5-WO_3/TiO_2-SO_4^{2-}$  CATALYSTS

The results of thermo stability for poisoned and recovered catalysts are presented in Fig.6. In the first stage before 100 °C, the evident weight loss of samples were resulted from the desorption of physical adsorbed water. For poisoned and recovered at 250 °C catalysts, apart from the weight loss result from H<sub>2</sub>O desorption, a new weight loss at the temperature between 200-400 °C were also observed. Such small weight loss could be attributed to the decomposition of ammonium sulfate (Nam I S, 1986). However, the corresponding loss was not detected in fresh and recovered at 490 °C samples, which illustrated that poisoned catalyst could achieve to the original fresh SCR activity after regenerated at 490 °C. The last weight loss between 700-900 °C for all the catalysts was corresponded to the sublimation of tungsten oxide (Jie Dang, 2013).

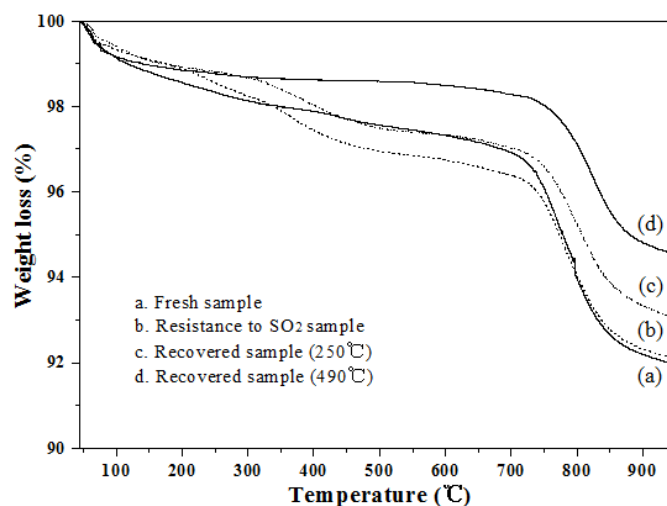


FIG. 6 TG ANALYSES OF POISONED V<sub>2</sub>O<sub>5</sub>-WO<sub>3</sub>/TiO<sub>2</sub>-SO<sub>4</sub><sup>2-</sup> CATALYSTS

## 4 CONCLUSIONS

The SCR activity between 120-220 °C of the V<sub>2</sub>O<sub>5</sub>-WO<sub>3</sub>/TiO<sub>2</sub>-SO<sub>4</sub><sup>2-</sup> catalyst was greatly improved by TiO<sub>2</sub> surface sulfated with 0.4M H<sub>2</sub>SO<sub>4</sub>. It could be affirmed that sulfuric acid doping had positive effect on low-temperature activity of the catalyst. In the presence of 300ppm SO<sub>2</sub> for 50h at 180 °C, the activity of V<sub>2</sub>O<sub>5</sub>-WO<sub>3</sub>/TiO<sub>2</sub>-SO<sub>4</sub><sup>2-</sup> catalyst dropped from 95% to 90%, which was always superior to that of V<sub>2</sub>O<sub>5</sub>-WO<sub>3</sub>/TiO<sub>2</sub>. The main deactivating substances were ammonium sulfate salts which deactivate the catalyst activity through pore plugging and covering. After calcined at 490 °C, poisoned catalyst could be recovered to the original levels along with ammonium sulfate salt decomposing. This result further ensured that there was no other metal sulfate which decomposed above 490 °C formed in the SCR reaction.

## ACKNOWLEDGMENT

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