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Study on Co-Effect of K_2SO_4 Deposition and Low Concentration SO_2 on Performances of V_2O_5/AC Catalysts for Low Temperature SCR

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Abstract

Simulated compounds were prepared by loading K_2SO_4 onto V_2O_5/AC catalysts. Study the effect of K_2SO_4 on V1/AC catalysts in the presence of low concentration SO_2. Transient response techniques, TPD was carried out. The results indicated that the DeNO activity of V1/AC catalysts was decreased seriously in the early period of operation, but the deactivation was gradually diminished with SO_2 adsorption and then, it was completely eliminated. For the sulphated catalysts (saturated catalysts by sulphate), their SCR activity were free from existence of gaseous SO_2, the loss of activity about 10% caused by K_2SO_4 was found on them. The deactivation of K_2SO_4 deposited catalysts was due to the decrease of adsorbed and activated NH_3, or some acid sites.

1. Introduction

Selective catalytic reduction of NO_x (SCR) is one of the most widely used technologies for reducing NO_x from stationary sources. The optimum operating temperature of the commercial catalyst (*i.e.*, V_2O_5/TiO_2 promoted with WO_3 or MoO_3) is in the range of 300˚C - 500˚C for keeping free from SO_2 deactivation, which makes it necessary to reheat the flue gas after the electrostatic precipitator and desulphuriser (120˚C - 250˚C). Thus the search for new catalysts which are active and stable at low temperatures and in presence of low SO_2 is still under way. In recent years, a considerable effort has been focused on some low temperature SCR catalysts such as MnO_x/NaY zeolite [1], MnO_x/activated carbon (MnO_x/AC) [2,3] and especially V_2O_5/AC [4-6].

There are some submicron particles (though very little) which contain alkali and alkali earth metals in coal fired flue gas especially in some coal-biomass co-fired flue gases even after the electrostatic precipitator and desulphuriser [7-9]. Those metals, mainly potassium, volatize from coal during combustion and then condense to form submicron particles such as K_2SO_4 or K_2O. They are major poisoning substances for SCR catalyst [10-12]. It is widely believed that the catalyst activity is related to the ammonia adsorbed on Bronsted acid sites associated with V^{5+}-OH [13-15]. When deposited, potassium preferentially co-ordinates to the hydroxyl groups on V_2O_5 and the catalytic activity decrease with the decrease in both the number and the strength of the Bronsted acid sites [10,12].

Under lower temperature (150˚C - 250˚C), V_2O_5/AC has been proved to be a promising catalyst for SCR, and also a perfect absorbance of SO_2 due to its catalytic oxidation of SO_2 to SO_3 [5,6,16]. More interestingly, the adsorption of SO_2 on V_2O_5/AC catalyst can remarkably promote the activity of SCR over it [17], which was suggestibly due to the increased acidity of the catalyst. When the V_2O_5/AC catalyst is used for SCR downstream of desulfurizer, there is also some low concentration of SO_2 in flue gas. In this case, the possible deactivation of V_2O_5/AC by deposition of potassium compounds is an important factor to be considered for the performance of this catalyst. However, there are few research have been reported to study it so far. This paper is to investigate the effect of potassium in flue gas on activity

of V_2O_5/AC catalyst for SCR in presence of low SO_2. K_2SO_4 was loaded on V_2O_5/AC catalyst as model compound of potassium by method of pore volume impregnation. The use of activity measurement, transient response techniques, and temperature programmed techniques (TPD) enable us to probe into the reason for the changes in activity of V_2O_5/AC under deposition of K_2SO_4 and sulfation in low SO_2.

2. Experimental

2.1. Catalyst Preparation

The activated carbon (AC) used was a commercial product from Shanxi Xihua Chem. Co. LTD. The AC was crushed into particles of 0.3 - 0.6 mm in diameter and subjected to proximate and ultimate analyses. V_2O_5 was supported to the AC by pore volume impregnation using an aqueous solution containing ammonium metavanadate and oxalic acid. The sample was then dried at 50˚C for 12 h and 110˚C for 5 h, followed by calcination in Ar at 500˚C for 8 h and pre-oxidation in air at 250˚C for 5 h. The catalyst used in this work which contains 1wt% V_2O_5 was termed as V1/AC.

K_2SO_4 was loaded onto V1/AC also through impregnation, followed by drying at 50˚C for 12 h and at 110˚C for 5 h. The K_2SO_4 loaded catalysts were termed as KxV1/AC, where x denoted the weight percentage of K in the catalyst. -S-SCR standed for the catalysts after SCR reaction in presence of low SO_2 at 250˚C till being saturated by sulphate and SCR activity keeps steady.

2.2. Catalytic Activity Measurement

SCR activity of the catalysts was measured in a fixed bed glass reactor of 15 mm in diameter. The feed rates of NH_3/Ar, NO/Ar and SO_2/Ar were controlled by mass flow meters and that of Ar and air were controlled by rotameters. H_2O vapor was introduced by passing the Ar stream through a heated gas-wash bottle containing deionized water. The overall feed contained 600 ppm NO, 600 ppm NH_3, 3.4% O_2, 2.6% H_2O vapor, 300 ppm SO_2 (when used) and balanced Ar. The total flow was maintained at 300 ml/min all the time, corresponding to a space velocity of 22500 - 4500 h^{-1} in the case of 0.5 - 2.5 g Cat. being used. The reaction temperature was varied between 100˚C - 250˚C. The concentration of NO and O_2 in the inlet and outlet of the reactor was measured on-line by a flue gas analyzer (KM9006). Conversion of NO, X_{NO}, is defined by (1)

$$X_{NO} = \frac{C_{in} - C_{out}}{C_{in}} \times 100\%$$ (1)

where C_{in} and C_{out} are NO concentrations in the inlet and outlet, respectively. $-dX_{NO}$, the differences of NO conversion between V1/AC with and without K_2SO_4 loaded, is used to denote the deactivation of V1/AC by loading K_2SO_4. In the experiment, the content of SO_2 adsorbed on V1/AC is calculated and labeled as SC (mg SO_2/g Cat.).

2.3. Transient-Response

Transient-response experiments have been widely used for the SCR study of NO over commercial V_2O_5/TiO_2 catalysts [18-20]. We carried out experiments at 250˚C by stepwise removal-addition one of the reactants SO_2, O_2 or NH_3 from steady-state. Contrarily to steady-state experiments, the dynamics of SCR reaction can be analyzed. Thus these experiments were allowed to unravel the steps involved in the mechanism of reaction. From the steady-state reaction conditions, the same as in 2.2, one reactant was replaced by equal volume of balance Ar, the outlet NO concentration was measured on-line by flue gas analyzer.

2.4. Temperature-Programmed Desorption (TPD)

The sulphate species in the catalyst with or without K_2SO_4 loaded were characterized by temperature-programmed desorption (TPD) with the use of a mass spectrometer (Balzers QMS422) for the gas stream analysis. TPD experiments were carried out in a quartz microreactor. In a typical experiment, 100ml/min of Ar was passed over the catalyst until the signal of the mass spectrometer was stable. Later the temperature was raised to 700˚C at a rate of 10˚C/min. The following main mass-to-charge (m/e) ratios were used to monitor the outlet gas composition: 17 (NH_3), 18 (H_2O), 28 (N_2/CO), 30 (NO), 32 (O_2), 44 (CO_2), and 64 (SO_2).

3. Results and Discussion

3.1. Catalytic Activity

It is widely believed that vanadium in oxidation state +5 undergoes a redox cycle and oxidizes SO_2 to SO_3 [16,21-23], and in the case of AC as supporter, the converted SO_3 companying with H_2O is retained by the AC supporter (sulphation of Cat.). **Figure 1** shows the real-time NO conversion along with the outlet SO_2 concentration as a function of reaction time on stream over V1/AC catalyst with or without K_2SO_4 loaded. Firstly, it can be observed that the SO_2 concentration describes a typical breakthrough curve. The SO_2 breakthrough time on each catalyst follows the order:
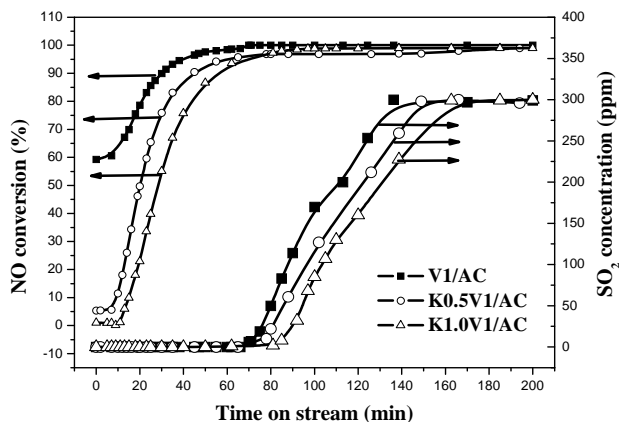
Figure 1. NO conversion and SO$_2$ released in experiment as a function of the reaction time on stream over V1/AC with or without K$_2$SO$_4$ loaded. Reaction conditions: 1.0 g catalyst, temperature 250°C, [NO] = 600 ppm, [NH$_3$] = 600 ppm, [SO$_2$] = 300 ppm, 3.4% O$_2$, 2.6% H$_2$O vapor, Ar to balance, total flow rate = 300 ml/min and SV = 11250 h^{-1}.

K1.0V1/AC > K0.5V1/AC > V1/AC, and the saturation time also follows the same order. At any time in this experiment, the SO$_2$ capacity (SC) of each catalyst can be calculated by area integration. Secondly, what should be focused on is the comparison of NO conversion over these three catalysts. Over V1/AC, the NO conversion is about 60% initially, and then increase after several minutes with the reaction goes on. After about 50 min of reaction, the NO conversion reaches to nearly 100% and keeps steady. However, over K$_2$SO$_4$ loaded catalysts, K0.5V1/AC and K1.0V1/AC, the initial NO conversions are very slow compared to V1/AC, about 6.0% and 0% respectively. Also after several minutes of reaction, they begin to increase with time, and reach to steady states at about 99%. From these curves, we can also observe that the NO conversion of K$_2$SO$_4$ loaded catalysts increase more rapidly than that of V1/AC. It seems that the deactivation of V1/AC for SCR by K$_2$SO$_4$ diminishes itself along with the reaction continues. Factually, the surface chemistry of the catalyst is continuously changed in this process along with more and more SO$_2$ is adsorbed, on the catalyst until saturated.

   Figure 2 shows the real-time relationship of NO conversion with SC over each catalyst. Apparently, NO conversion increases with the increase of SO$_2$ adsorbed either on V1/AC or K$_2$SO$_4$ loaded V1/AC. The difference is that, on K$_2$SO$_4$ loaded V1/AC, NO conversion increases more drastically, in other word, the SCR activity is more promoted by sulphation. Notably, to reach to the initial NO conversion of V1/AC, there should be 5.8 mg SO$_2$/g Cat. and 8.0 mg SO$_2$/g Cat. adsorbed on K0.5V1/AC and K1.0V1/AC respectively. It suggests that the deactivation of V1/AC by 0.064
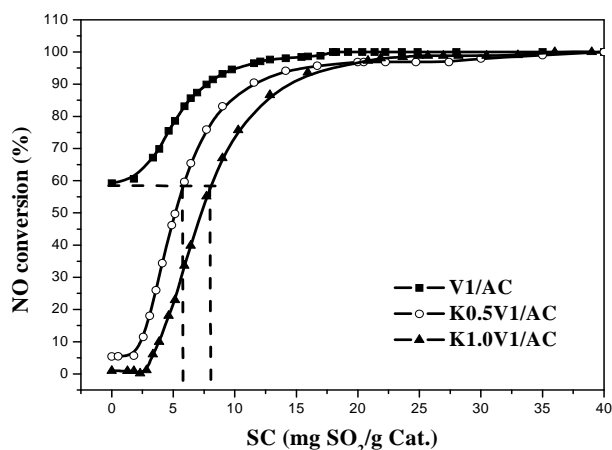

Figure 2. NO conversion on-line as a function of the content of SO$_2$ adsorbed (SCR) on catalyst V1/AC, K0.5V1/AC and K1.0V1/AC.

mmol/g and 0.128 mmol/g K$_2$SO$_4$ can be counteracted by adsorption of 0.091 and 0.125 mmol/g SO$_2$ respectively. The deactivations of V1/AC by 0.5% and 1.0% K as a function of SC are calculated as showes in **Figure 3**. -dX$_{NO}$ as a sign lost activity is calculated from the difference of NO conversion between catalysts with and without K$_2$SO$_4$ loaded. Along with the SC increases, the lost activities caused by 0.5% and 1.0% K decrease from the initial (SC = 0) 54% and 60% to 0% as the SC increases to about 25 mg SO$_2$/g Cat. and then, the deactivation is completely eliminated. In this period, V1/AC catalyst is more deactivated by 1.0% K than 0.5% K.

3.2. Effect of Temperature and Space Velocity

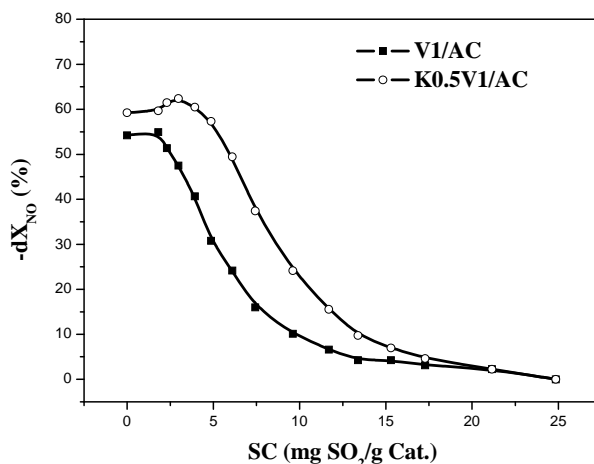**Figure 4** shows the steady NO conversion of sulphated

Figure 3. Deactivation of V1/AC by 0.5% and 1.0% potassium loaded in form of K$_2$SO$_4$ as a function of SO$_2$ adsorbed.
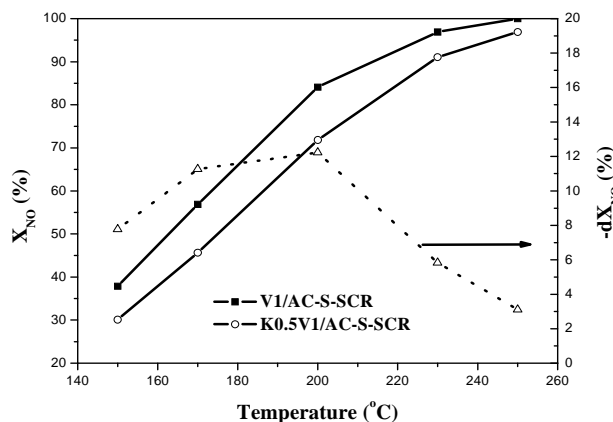
Figure 4. Steady NO conversion of sulphated V1/AC and K0.5V1/AC as a function of reaction temperature. Reaction conditions: 0.5 g catalysts S-SCR, temperature 150˚C - 250˚C, [NO] = 600 ppm, [NH₃] = 600 ppm, [SO₂] = 300 ppm, 3.4% O₂, 2.6% H₂O vapor, Ar to balance, total flow rate = 300 ml/min and SV = 22500 h⁻¹.

V1/AC and K0.5V1/AC as a function of reaction temperature in the temperature range of 150˚C - 250˚C. The used catalysts are V1/AC and K0.5V1/AC after 180 min reaction under the conditions in **Figure 1**, and thus they are saturated by S, namely sulphated. At each temperature, NO conversion increases from start time to reach a steady state, namely steady NO conversion. It can be observed that the steady NO conversion of either V1/AC or K0.5V1/AC increases with the reaction temperature, which seems to be a symptom of reaction limited step. In the whole temperature range of 150˚C - 250˚C, the steady NO conversion of K0.5V1/AC keeps lower than that of V1/AC, indicating that a considerable deactivation of V1/AC has been caused by loading K_2SO_4 at a higher space velocity (SV = 22500 h⁻¹) even when sulphated. The difference in NO conversion between K0.5V1/AC and V1/AC is from 3.2 % at 250˚C to the maximum 12.2% at 200˚C, suggesting that the V1/AC is mostly deactivated by K_2SO_4 at 200˚C. Apparently, raising the reaction temperature can not only improve the SCR activity of sulphated V1/AC but also change the deactivation by the poisonous K_2SO_4.

The SCR activities of sulphated V1/AC and K0.5V1/AC at varied space velocity (SV) are also measured at 200˚C, the results are compared in **Figure 5**. Over V1/AC, NO conversion keeps 100% when SV is between 4500 h⁻¹ and 5625 h⁻¹, then comes to decrease along with the SV increasing, till the SV achieves 22,500 h⁻¹, the NO conversion decreases to 84.13%. While over K0.1V1/AC, NO conversion directly decreases from 100% to 72.05% with the SV increases from 4500 h⁻¹ to 22,500 h⁻¹. In Comparison, the increasing of space velocity draws more negative effect on K0.5V1/AC than V1/AC. In other word, the load of K_2SO_4 deactivates the sulphated V1/
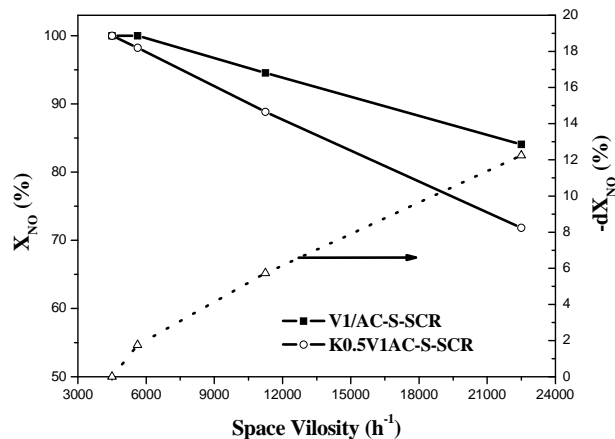

Figure 5. Steady NO conversion of sulphated V1/AC and K0.5V1/AC as a function of space velocity (SV). Reaction conditions: catalysts S-SCR, temperature 200˚C, [NO] = 600 ppm, [NH₃] = 600 ppm, [SO₂] = 300 ppm, 3.4% O₂, 2.6% H₂O vapor, Ar to balance, total flow rate = 300 ml/min and SV = 4500 - 22500 h⁻¹.

AC more seriously at higher space velocity. It implies that some active centers with higher transform frequency may be poisoned by K_2SO_4.

3.3. Transient-Response Reactions

Figure 6 shows experiments with stepwise removal of SO₂ and O₂, and then, O₂ is added to V1/AC-S-SCR and K0.5V1/AC-S-SCR respectively, from the steady state of SCR in low SO₂. Initially, the steady NO conversions of V1/AC and K0.5V1/AC are 100% and 99%. When SO₂ is removed, there is no change in NO conversion of two
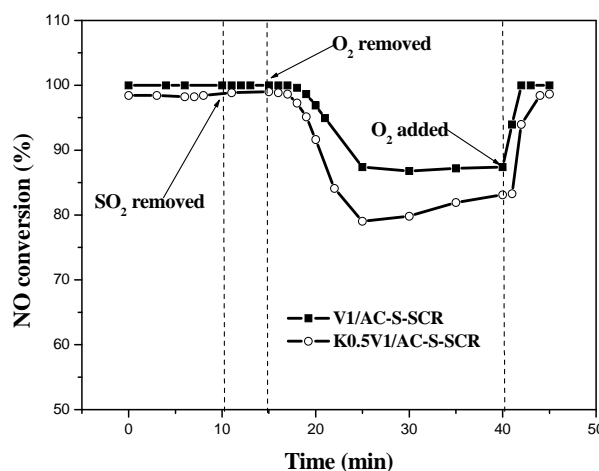

Figure 6. NO concentration upon stepwise removal-addition of SO₂ and O₂ in the SCR of NO in presence of low SO₂ over sulphated V1/AC and K0.5V1/AC. Reaction conditions: 1.0 g catalyst, temperature 250˚C, [NO] = 600 ppm, [NH₃] = 600 ppm, [SO₂] = 300 ppm, 3.4% O₂, 2.6% H₂O vapor, Ar to balance, total flow rate = 300 ml/min and SV = 11250 h⁻¹.

catalysts. It implies that sulphated catalysts can maintain high SCR activity in absence of gaseous SO_2. In other word, catalyst is promoted by adsorbed SO_2 but not gaseous SO_2. When O_2 is removed, the NO conversion of V1/AC continues to be 100% for about 4min and then declines slowly to 85% and keeps steady. Similar tendency is found on K0.5V1/AC, excluding that the steady NO conversion in absence of O_2 is lower than that on V1/AC. After the recovery of O_2, the NO conversion of either V1/AC or K0.5V1/AC refreshed to initial value rapidly. From this figure, some deductions can be drawn: Firstly, SCR activity of V1/AC catalyst is free from existence of gaseous SO_2, but is promoted by sulphation of the catalyst. Secondly, on sulfated catalyst, SCR reaction can process in absence of O_2 after the adsorbed O_2 is consumed. Adsorbed O_2 can also heighten its activity remarkably as well as gaseous O_2, which suggests the below reaction occurs:

$$6NO + 4NH_3 \rightarrow 5N_2 + 6H_2O \qquad (2)$$
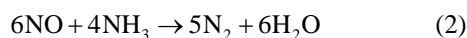
Thirdly, the deposition of K_2SO_4 on sulfated catalyst restrains the above reaction by a 10 percents decrease in NO conversion. In other word, the absence of O_2 enhances the deactivation of catalyst by K_2SO_4.

Figure 7 shows the experiments with stepwise removal-addition of NH_3 to the sulphated V1/AC and K0.5V1/AC respectively from steady reaction. After the removal of NH_3 from the steady state, NO conversions of V1/AC and K0.5V1/AC continue to be about 100% for 23 min and 18 min respectively, and then come to decrease. After 45 min, NO conversion of each catalyst reaches bottom value about 10%. Apparently, NH_3 reacts through a strongly adsorbed state, which is in accordance with previous report [19]. Otherwise, the existence of K_2SO_4 has considerably decreased the reactive NH_3 adsorption on sulphated V1/AC.

According to the results obtained from **Figure 6** and **Figure 7**, it is clearly that the SCR reaction over sulfated V1/AC either with K_2SO_4 loaded or not visibly relates to O_2 and NH_3. Thus the NO conversion after the simultaneous removal of O_2 and NH_3 from steady state is measured as showed in **Figure 8**. Compared to **Figure 7**, the times for NO conversion remaining at 100% over V1/AC and K0.5V1/AC drastically decrease to 8 min and 3 min respectively. In two cases, the step-wise experiment both start from steady SCR state, so the adsorbed NH_3 on a certain catalyst sample is equal. The difference is that the gaseous O_2 was removed or not. Obviously, gaseous O_2 is important for the reaction of adsorbed NH_3. In absence of O_2, only a little part of adsorbed NH_3 can react with NO but most of it can not. In other word, most of the sites for NH_3 adsorption are not active for SCR in absence of O_2. Comparatively, those O_2-free active sites on
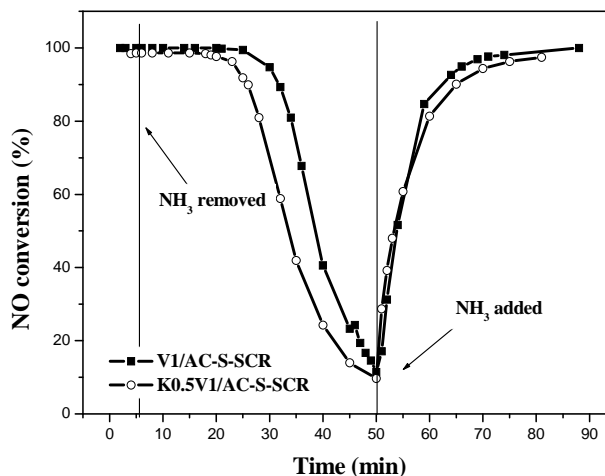

Figure 7. NO concentration upon stepwise removal-addition of NH_3 in the SCR of NO in presence of low SO_2 over sulphated V1/AC and K0.5V1/AC. Reaction conditions: 1.0 g catalyst, temperature 250°C, [NO] = 600 ppm, [NH_3] = 600ppm, [SO_2] = 300 ppm, 3.4% O_2, 2.6% H_2O vapor, Ar to balance, total flow rate = 300 ml/min and SV = 11250 h^{-1}.
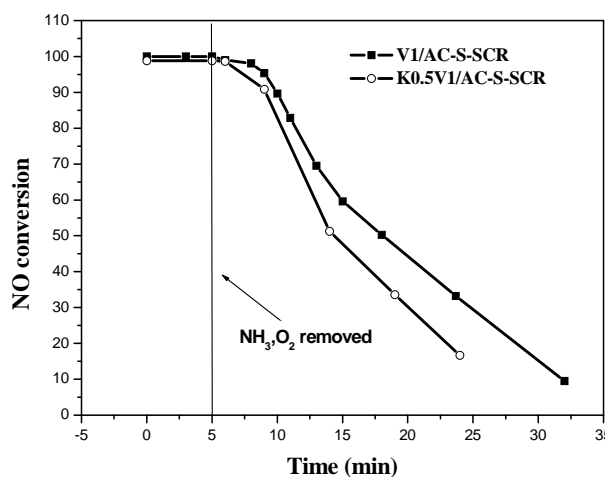

Figure 8. NO concentration upon removal of NH_3 + O_2 in the SCR of NO in presence of low SO_2 over sulphated V1/AC and K0.5V1/AC. Reaction conditions: 1.0 g catalyst, temperature 250°C, [NO] = 600 ppm, [NH_3] = 600 ppm, [SO_2] = 300 ppm, 3.4% O_2, 2.6% H_2O vapor, Ar to balance, total flow rate = 300 ml/min and SV =11250 h^{-1}.

V1/AC are more than on K0.5V1/AC, which is in accordance with the results in **Figure 6**.

3.4. Temperature-Programmed Desorption of SO_2 and NH_3

In this work, it is obviously that both the loading of K_2SO_4 and the adsorption of SO_2 on V1/AC catalyst have considerably affected its SCR activity, which is generally recognized to be related to the adsorption and

activation of NH$_3$ on the catalyst. Thus, TPD experiments were carried out on V1/AC-S-SCR and K0.5V1/AC-S-SCR to examine the desorption of SO$_2$ and NH$_3$ from each sample as shown in **Figure 9** and **Figure 10** respectively. **Figure 9** shows the similar SO$_2$ TPD profiles of V1/AC-S-SCR and K0.5V1/AC-S-SCR. The desorption of SO$_2$ from each sample starts at about 290˚C and reached the first (and also the main) peak at about 345˚C. Along with the temperature increasing, a shoulder peak appears after 400˚C for both samples. It is apparently that SO$_2$ desorbs from K0.5V1/AC-S-SCR is more than that from V1/AC-S-SCR, which is in accordance to the result of **Figure 1**. In **Figure 10**, both the V1/AC-S-SCR and K0.5V1/AC-S-SCR appear a main NH$_3$ desorption peak at about 315˚C with similar profile. This peak may assign to the NH$_3$ desorption from acid sites which can be independent from alkali metal and adsorbed SO$_2$, so the peak profile and intensity is similar. Also, there aresome notable differences in NH$_3$ TPD profiles of the two samples. Besides the first main peak,
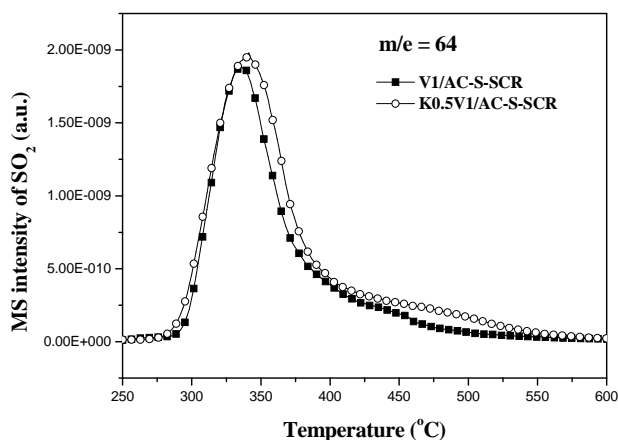
another two desorption peaks at about 450˚C and 550˚C are observed on V1/AC-S-SCR,but can't be found on K0.5V1/AC-S-SCR. These two peaks are related to NH$_3$ desorption from acid sites which inclined to be affected by K ion. When K is addition to the V1/AC-S-SCR catalyst, the acidity of the two acid sites decrease, in other words, NH$_3$ adsorption capacity decrease, so the deactivation take place. More SO$_2$ adsorption on K0.5V1/AC-S-SCR (**Figure 2**) may enhance the acidity of the two acid sites, so the deactivation can be counteracted.

4. Conclusions

The deposited K$_2$SO$_4$ decrease the SCR activity of V$_2$O$_5$/AC catalyst, but the deactivation caused by K$_2$SO$_4$ can be counteracted by SO$_2$ adsorption. The SCR activity of both V1/AC and KxV1/AC at low temperature (150˚C - 250˚C) can be promoted by SO$_2$ adsorption especially the latter. The deactivation caused by K$_2$SO$_4$ can be enhanced with space velocity increasing but being counteracted with temperature increasing. When the catalysts are saturated and steady, their SCR activity is no longer affected by the gaseous SO$_2$. And their SCR process can still process without gaseous or adsorbed O$_2$, but the absence of O$_2$ enhances the deactivation of catalyst by K$_2$SO$_4$. The deactivation of K$_2$SO$_4$ deposited catalysts is due to the decrease of adsorbed and activated NH$_3$, or some acid sites.

5. Acknowledgements

Figure 9. SO$_2$ TPD profiles of V1/AC-S-SCR and K0.5V1/ AC-S-SCR.
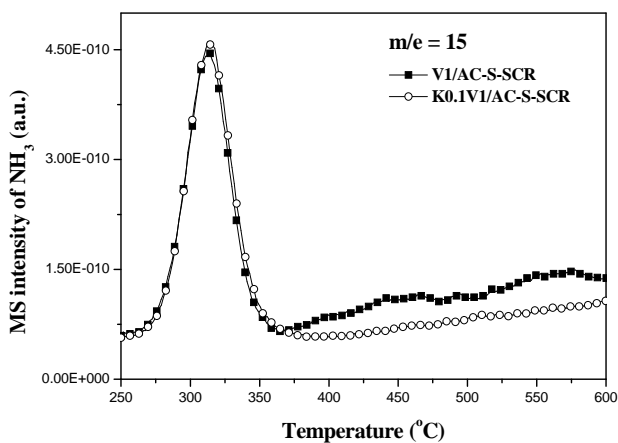
Figure 10. NH$_3$ TPD profiles of V1/AC-S-SCR and K0.5V1/ AC-S-SCR.

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