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RESEARCH ARTICLE

Effect of pre-oxidation process on V_2O_5/AC catalyst for the selective catalytic reduction of NO_x with NH_3

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Abstract

Activated coke-based catalysts have attracted extensive attention in denitration by selective catalytic reduction by NH_3 (NH_3-SCR), due to their excellent catalytic performance at low temperature. In the paper, the V_2O_5/AC catalyst was prepared by the impregnation method to investigate the effect of pre-oxidation process on its NH_3-SCR activity. Activity test results show that the V_2O_5/AC catalyst with 4-h pre-oxidation exhibits the best NO_x removal efficiency, which reaches the NO_x conversion is over 75% in the range of 200–240 °C and exhibits an excellent resistance to SO_2 and H_2O. Characterization results demonstrate that the V^{4+} was oxidized by oxygen molecule during pre-oxidation process, which contributes to the formation of V^{5+} ions and surface-active oxygen species. The surface-active oxygen species are conducive to promoting the "fast SCR" reaction; thus, the pre-oxidized process can contribute to the superior NH_3-SCR performance for V_2O_5/AC catalyst at low temperature.

Keywords NH_3-SCR · Activated coke · V_2O_5/AC · Pre-oxidation

Introduction

The use of fossil energy produces a large number of nitrogen oxides, which could cause acid rain, photochemical smog, and harm to human health (Hou et al. 2014; Jiang et al. 2016; Tang et al. 2016). With the implementation of ultra-low emission standard for atmospheric pollutants in China, the promotion and implement of flue gas denitrification (De-NO_x) technologies will be forced and stressed (Xie et al. 2019). The selective catalytic reduction of NO_x by NH_3 (NH_3-SCR) is a mature technology to solve NO_x emissions. The catalyst is essential for NH_3-SCR technology, and it determines the operating temperature and cost of the industrial SCR system. V_2O_5-WO_3/TiO_2 is extensively used as the SCR denitration catalyst due to its high catalytic activity and resistance to SO_2 poisoning. However, the active window temperature of V_2O_5-WO_3/TiO_2 catalyst is higher (320–400 °C), and it is not suitable for low-temperature flue gas (below 150 °C) in iron and steel, cement, glass, and other industries (Yang et al. 2020). Therefore, it is important to research a highly efficient NH_3-SCR catalyst at low temperature. Some catalysts show a high SCR activity in the range of 120–250 °C, such as MnO_x/Al_2O_3, CuO/Al_2O_3, and Mn/TiO_2 catalyst, but their catalytic activities easily are inhibited by SO_2(Hou et al. 2014).

Developing a catalyst with high resistance to SO_2 deactivation at low temperature is critical to the NH_3-SCR process. Studies have shown that it is superior to TiO_2 and SiO_2 in terms of resistance to ammonium sulfate, activated carbon, or coke as a carrier (Han et al. 2018). Compared with the activated carbon, activated coke (AC) has the characteristics of low cost and high mechanical strength (Ye et al. 2020). AC has a large specific surface area, abundant surface functional groups, and strong adsorption capacity, which had been successfully applied to SO_2 and Hg capture and NO_x purification of flue gas in the steel industry (Liu and Liu 2013, Li et al. 2018, Li et al. 2019, Ye et al. 2020). Pure AC shows a low catalytic activity; thus, the method of introducing new active components (such as MnO_x, CuO, or V_2O_5) is used to enhance its denitration activity (Marban et al. 2004; Boyano et al.

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2008; Lazaro et al. 2009). Compared with these catalysts, AC-supported V_2O_5 (V_2O_5/AC) catalysts are more promising, because those still have good catalytic activity in the presence of low moisture and SO_2 conditions (Sun et al. 2009; Wang et al. 2014). In order to further develop the V_2O_5/AC catalyst, the studies about NH_3-SCR reaction mechanism and active site were carried, and the result has shown that the V_2O_5 species are the main sites for the adsorption and activation of NH_3(Sun et al. 2009).

AC has characteristic of flammable at high temperature; thus, N_2 atmosphere is used during loading V_2O_5 species (the decomposition of NH_4VO_3 into V_2O_5 occurs only when the temperature is over 500 °C). After loading V_2O_5 species, and calcined (pre-oxidized) under air atmosphere to activate the V_2O_5 and catalyst. According to the literature, pre-oxidized catalyst can enhance catalytic activity, compared with the non-pre-oxidized catalyst, and the pre-oxidized catalyst has better catalytic activity and stability (Zhu et al. 1999). However, there is no literature report on the relationship between pre-oxidation process and catalytic activity. In this work, catalysts were prepared by different pre-oxidation time and tested for their NH_3-SCR performance at low temperature, and the SEM, BET, XPS, H_2-TPR, and NH_3-TPD characterizations were performed to explore the reasons for this phenomenon and construct the structure-activity relationship of the catalyst.

Experimental

Catalyst preparation

The V_2O_5/AC catalyst was prepared by the impregnation method. Generally, 5 g activated coke (40–80 mesh) and 0.3214 g NH_4VO_3(AR) were added into 100 mL deionized water under vigorously stirring for 1 h at room temperature. Subsequently, the mixture solution was stirred at 80 °C for 2 h followed by drying at 120 °C for 5 h and then calcined at 400 °C in a N_2 atmosphere for 5 h to obtain a V_2O_5/AC catalyst. After that, the catalyst was pre-oxidized at 250 °C, the obtained V/AC catalysts was labeled as V/AC-X (X was pre-oxidization time, X=0, 3, 4, and 5). Among them, V/AC-0 means the preparation of the V_2O_5/AC catalyst without pre-oxidation.

Characterization

The Supplementary materials provide the particulars of characterization.

Catalyst activity tests

The efficiency of NO_x catalytic removal was tested in a fixed-bed reactor (i.d. 7mm). The mass of catalyst was 0.7 g (40–80 mesh) in the test, the feeding gases were mixed by the gas mixer and then sent to the reactor, the total flow rate was 200 mL/min, and the inlet gas hourly space velocity (GHSV) was maintained at 12000 h^{-1}. The inlet gas concentrations of NO and NH_3 in the feeding gas both were 500 ppm, while O_2 was 5%, and the N_2 was balanced gas. Meanwhile, 5 vol % H_2O (when used), 300 ppm SO_2 (when used) was added in the test. The values of NO_x removal efficiency can be calculated by following equation:

$$NO_x \text{ removal efficiency } (\%) = \left([NO_x]_{in} - [NO_x]_{out}\right)/[NO_x]_{in} \times 100\%$$

Results and discussion

Catalyst performance

Fig. 1 exhibited the NO_x removal efficiency of AC and V/AC-X (X= 0, 3, 4, and 5) catalysts. The result indicated that the NO_x conversion decreased in the following order: V/AC-4 > V/AC-3> V/AC-5> V/AC-0>AC. The AC exhibited a low activity, the NO_x conversion at 120 °C was only 20%, and it reduced with the increase of reaction temperature. This result was similar with research previously (Jing et al. 2014). After loading V_2O_5 species, the NO_x removal efficiency of the V/AC catalyst increased significantly, suggesting that V_2O_5 is the main active species of NH_3-SCR reaction. After pre-oxidizing, the catalytic activities had been further improved; for the V/AC-0, the NO_x conversion was 76% at 240 °C, while V/AC-X were 85% (V/AC-3) and 94% (V/AC-4). However, the NO_x conversion of V/AC-5 was 83% at 240 °C, which was lower than V/AC-4. In summary, the pre-oxidation process can contribute to the superior NH_3-SCR performance of V/

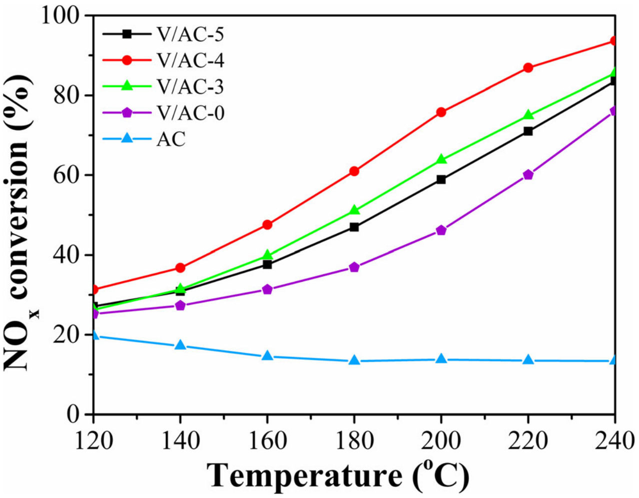

Fig. 1 NO_x conversion of samples at different temperatures

AC-X catalyst, and pre-oxidation time is the most important influencing factor; the best pre-oxidize time is 4 h.

The long-term stability of V/AC-4 catalyst was investigated by testing the NH_3-SCR performance of the catalyst at 240 °C for 60 h. The result was exhibited in Fig. S1a. The NO_x removal efficiency of the catalyst was not decreased within 60 h, but increased a little (the NO_x conversion has increased from 94.0 to 97.7%), suggesting that the stability of V/AC-4 catalyst was good. The effect of SO_2 and water vapor on V/AC-4 catalyst was examined at 240 °C. As presented in Fig. S1b–c, when 300 ppm SO_2 and 5 vol% H_2O were added, respectively, the NO_x removal efficiency of V/AC catalyst increases and keeps the increase trend slightly. After removing SO_2 and H_2O, the catalytic activity of V/AC-4 is still increased with time, indicating that the tolerance of SO_2 and H_2O over V/AC-4 catalyst was good. After removing SO_2, the catalytic activity is still increased with time. The reason for the increased activity could be caused by the sulfate formed during test process, which enhanced the surface acidity of catalyst (Zhu et al. 2001). The more surface acidity of the catalyst could be beneficial to the adsorption and activation of ammonia, thereby increasing the NH_3-SCR activity. In general, water vapor and reaction gas produce competitive adsorption on the catalyst surface (Hou et al. 2014; Lu et al. 2018), thereby inhibiting the adsorption of reactive species and reducing the NH_3-SCR performance. Due to the hydrophobicity of the activated coke (Zhang et al. 2020), water vapor cannot adsorb on the catalyst surface, explaining why the V/AC-4 catalysts showed good H_2O resistance.

Catalyst characterization

In order to explain the influence of pre-oxidation process on the crystal structures of the catalyst, the AC and V/AC-X catalysts were characterized by XRD, respectively. The results are presented in Fig. 2. Two broad diffraction peaks located at 20–30° and 40–50° are characteristic diffractions of activated coke graphite crystals (Han et al. 2018; Yang et al. 2020). The characteristic peaks of SiO_2 (an additive substance used in the industrial production of activated coke) located at 20.8°, 26.6°, 36.6°, 44.0°, 50.1°, 60.0°, and 68.2° were observed (Ge et al. 2019). No peaks that belong to V_2O_5 species were detected in the patterns of V/AC catalyst, suggesting that vanadium species has extremely high dispersibility on the surface of AC carrier.

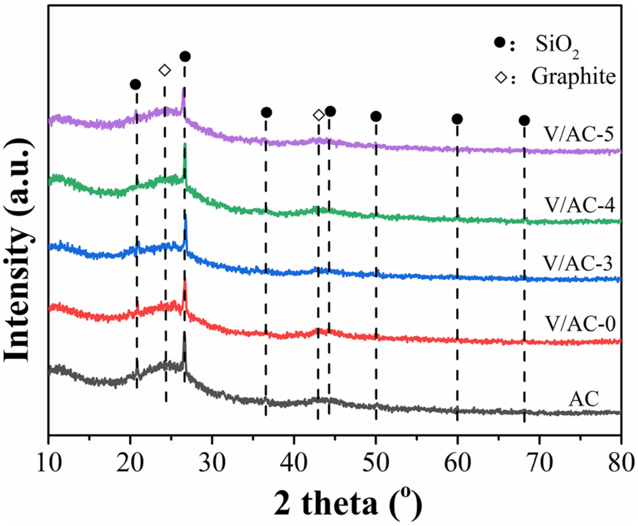

Fig. 2 XRD patterns of catalysts

The physical structures of AC and V/AC-X catalysts are presented in Table 1. AC exhibited a specific surface area of 214 $m^2\ g^{-1}$ and a total pore volume of 0.025 $cm^3\ g^{-1}$ with an average pore diameter of 4.44 nm. Compared with AC catalyst, the BET surface area, pore volume, and average pore size of the V/AC-0 catalyst decreased. With the increase of the pre-oxidation time, the specific surface area, pore volume, and average pore size of the V/AC-3 and V/AC-4 catalysts both increased, while the V/AC-5 catalyst is greatly reduced, which is consistent with activity test results, which due to the structure of V/AC-X catalyst changed during the pre-oxidation process, thereby affecting the catalytic activity. Compared with the V/AC-0 catalyst, the surface area of V/AC-5 catalyst is smaller, while the catalytic activity is better, indicating that the specific surface area is not the main factor for the enhancement of the catalytic activity. The specific surface area of the V/AC-4 catalyst is similar to the V/AC-3 and V/AC-0 catalyst, while the pore volume and average pore size of V/AC-4 catalyst was the largest, and it had the best NH_3-SCR performance, thus indicating that the new pore structure is formed to facilitate the NH_3-SCR reaction. In order to further explore the impact of the pre-oxidation process on the catalyst structure, the sample morphology and particle size were analyzed by SEM, and the results are presented in Fig. S2. Compared with AC catalyst, the surface of V/AC-0 catalyst is uneven (Fig. S2a–b), and it has shown that the structure of the catalyst was changed after loading vanadium species. With the increase of the pre-oxidation time, there was agglomerative phenomenon accompanying with V/AC-3 and V/AC-4 catalyst (Fig. S2c–e), while the V/AC-5 catalyst presents a number of large block crystals, obviously, which is consistent with the previous BET

Table 1 Specific surface area, pore volume, and pore diameter of the catalysts

Samples	Surface area ($m^2\ g^{-1}$)	Pore volume ($cm^3\ g^{-1}$)	Pore size (nm)
AC	214	0.025	4.44
V/AC-0	186	0.018	4.07
V/AC-3	197	0.028	4.86
V/AC-4	196	0.030	5.08
V/AC-5	166	0.018	3.93

Table 2 Element composition percentage

Samples	V^{4+}/ (V^{4+}+V^{5+})/%	O_α/(O_α+ O_β+ O_γ)/%	O_γ/(O_α+ O_β+ O_γ)/%
V/AC-0	60.6	20.88	5.45%
V/AC-3	43.3	48.22	14.61%
V/AC-4	30.6	49.05	19.90%
V/AC-5	29.0	48.32	21.10%
V/AC-4-used	32.4	49.32	20.08%

results. This phenomenon can explain the reason for that the NO_x removal efficiency of V/AC-5 catalyst is lower than that the V/AC-4 catalyst. Both results of SEM and BET tests have shown that the new pore structure formed in the catalyst surface during the pre-oxidation process, thus affecting their SCR performance at low temperature.

The surface elemental compositions of AC, V/AC-X, and V/AC-4-used (after 60-h stability test) catalysts were tested by XPS. The $V2p_{1/2}$ XPS results are exhibited in Fig. 3a. The peaks at 515.7–517.0 eV and 517.0–518.0 eV were attributed to V^{4+} and V^{5+}, respectively (Cha et al. 2013). The content of V^{4+} on the catalyst was calculated by the peak area $V^{4+}/(V^{4+}+V^{5+})$. As presented in Table 2, the content of V^{4+} on V/AC-0, V/AC-3, V/AC-4, and V/AC-5 catalysts was 60.6%, 43.3%, 30.6%, and 29.0%, respectively. With the increase of the pre-oxidation time, the V^{4+} content decreases, which indicated that the V^{4+} was oxidized to V^{5+} by the oxygen in air atmosphere (Zhu et al. 1999). After stability testing, the V^{4+} content of V/AC-4 sample increased from 30.6% (V/AC-4-fresh) to 32.4% (V/AC-4-used), which is due to the reduction of V^{5+} into V^{4+} by NH_3 during NH_3-SCR reaction.

The O1s XPS spectra are exhibited in Fig. 3b. The peaks at 529–530 eV, 531–533 eV, and 533–535 eV were attributed to lattice oxygen (marked as O_β), surface chemical oxygen (marked as O_α), and the oxygen species in hydroxide groups (marked as O_γ), respectively (Jiang et al. 2019). The proportions of $O_\alpha/(O_\alpha+ O_\beta+ O_\gamma)$ in V/AC-0 sample was 20.88%.

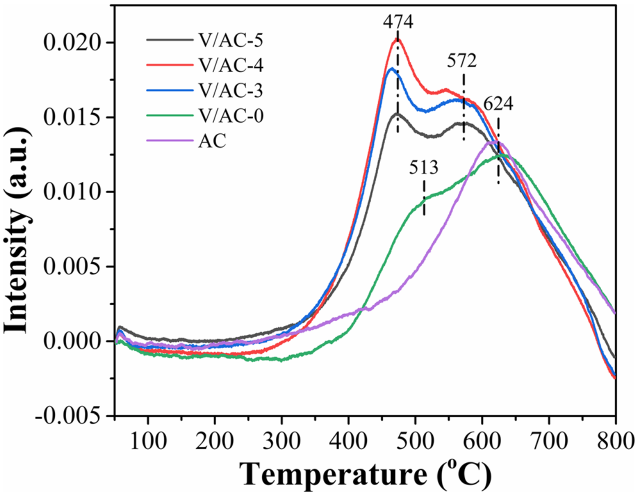

Fig. 4 H_2-TPR spectra of catalysts

After pre-oxidizing, the content of O_α is significantly increased. The content of O_α of V/AC-3, V/AC-4, and V/AC-5 catalysts were 48.22%, 49.05%, and 48.32%. According to previous reports, chemically adsorbed oxygen can boost that the NO was oxidized to NO_2, which can promote "fast SCR" reaction (Ma et al. 2011). After pre-oxidizing, the V^{4+} content decreased, and the O_α content increased, indicating that the V^{4+} was oxidized by oxygen molecule during pre-oxidation process, which contributes to the formation of V^{5+} ions and surface-active oxygen species, thereby improving the catalytic activity. Compared with the V/AC-4 catalyst, the O_α content and V^{4+} content of V/AC-4-used sample were both increased in the test, indicating that the O_α was formed during the V^{4+} and was oxidized to V^{5+}, while V^{4+} was generated again in NH_3-SCR reaction atmosphere, which can enhance the cycle of V^{4+}/V^{5+}, which explains why the catalytic activity increased gradually in the stability test. With the increase of the pre-oxidation time, the O_γ content increases, and this may be due to the formation of hydroxyl functional groups during the pre-oxidation process. The catalytic activity of the V/AC-3 catalyst is better than the V/AC-5 catalyst, which is

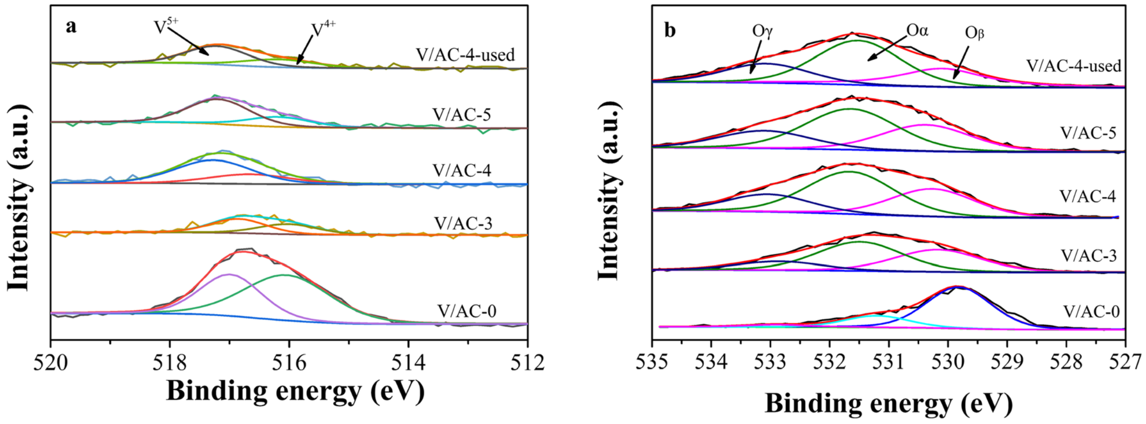

Fig. 3 The XPS spectra of catalysts: **a** V $2p_{1/2}$ and **b** O 1s

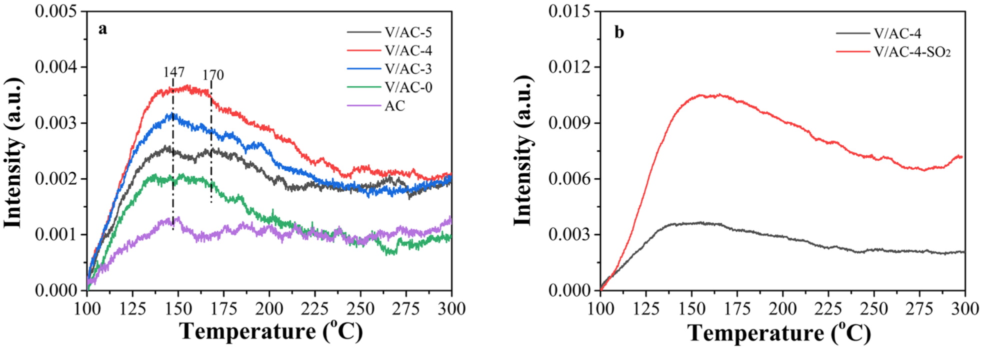

Fig. 5 NH_3-TPD spectra of catalysts

different from the activity results, thereby the content of O_γ is not the influencing factor of the catalytic activity.

The redox property of catalyst is one of the important influencing factors in SCR reaction. Fig. 4 exhibited the H_2-TPR curves of AC and V/AC-X catalysts. The peak located at 624 °C for AC catalyst was attributed to the vaporization peak of carbon carrier on activated coke (Zhang et al. 2020). The peaks located at 624 °C and 513 °C in the V/AC-0 catalyst were caused by the vaporization peak of carbon carrier and reduction peak of V^{5+}, respectively (Han et al. 2018). Compared with the AC catalyst, the reduction peak (after pre-oxidation) of V/AC-X catalyst shifted to a lower temperature, indicating that the interaction between activated coke carrier and vanadium species could improve the redox property of catalysts (Lu et al. 2018). With the increase of pre-oxidation time, the intensity of the reduction peak of V^{5+} and the vaporization peak of the activated coke carrier of the V/AC-3 and V/AC-4 catalyst are obviously enhanced, indicating that the content of V^{5+} and the redox sites of the catalyst are both increases during the pre-oxidation progresses. However, the reduction peak area of the V/AC-5 catalyst is significantly reduced. Combined with the result of BET, it could be shown that the excessive pre-oxidation time causes the sintering of active species on V/AC-5 catalyst, thereby decreasing its catalytic activity.

NH_3-TPD tests are performed to study the ammonia desorption over AC, V/AC-X, and V/AC-4-SO_2 (after sulfur resistance test) samples. As shown in Fig. 5a, the AC curve showed only one peak at 147 °C, while the V/AC-X samples present two broad peaks; the desorption peaks located at 147 °C and 170 °C were the physical adsorption of ammonia and the adsorption of NH^{4+} at the Brønsted acid site (Yin et al. 2018). After loading vanadium species, the desorption peak area increased significantly, indicating that vanadium species can provide more acidic sites on the catalyst surface, which can promote the adsorption and activation of ammonia. Compared with V/AC-0 catalyst, the desorption peak areas of the V/AC-3 and V/AC-4 further increase, suggesting that the pre-oxidation process promotes the adsorption of ammonia and can promote the formation of V^{5+} on the catalyst surface (Xu et al. 2018), which is consistent with the H_2-TPR results. However, the desorption peak area of the V/AC-5 catalyst is reduced, which is because catalytic activity species was agglomerated, and the adsorption of NH_3 is inhibited. As shown in Fig. 5b, NH_3-TPD patterns were performed among V/AC-4-SO_2 (after sulfur resistance test) samples, the desorption peak area of V/AC-4-SO_2 sample is larger, and this is because the sulfate formed during test process could improve the adsorption and activation of ammonia, thereby increasing the NH_3-SCR activity.

Conclusion

The activity test results showed that the NO_x catalytic removal efficiency of V_2O_5/AC catalyst is better than the AC catalyst, suggesting that the V_2O_5 is the active substance. The activity sequence of V/AC-X catalysts prepared by different pre-oxidation time at 120–240 °C is V/AC-4> V/AC-3> V/AC-5> V/AC-0. The results showed that the pre-oxidation process is beneficial to catalytic performance of V_2O_5/AC-X catalysts. Series of characterizations were used to explain the reasons for the phenomenon. The BET and SEM characterization results demonstrated that the specific surface area, pore volume, and average pore size of the catalyst increase with the increase of the pre-oxidation time, and the new pore structure is formed to facilitate the NH_3-SCR reaction. During the pre-oxidation progress, the V^{4+} was oxidized by oxygen molecule, which contributes to the formation of V^{5+} ions and surface-active oxygen species. The surface-active oxygen species is conducive to promoting the "fast SCR." In summary, the pre-oxidized V_2O_5/AC has the best catalytic activity when the pre-oxidation time is 4 h.

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Author contribution Jiahua Xie: Conceptualization, data curation, methodology, validation, writing - original draft.
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Zihua Wu: Conceptualization, methodology, writing - review and editing

Yiqing Zeng: Conceptualization, methodology, writing - review and editing

Shule Zhang: Funding acquisition, project administration, resources, supervision

Jing Liu: Funding acquisition, project administration, resources, supervision

Qin Zhong: Funding acquisition, project administration, resources, supervision

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Conflict of interest The authors declare no competing interests.

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