

Research Article

SO₂ Poisoning Behaviors of Ca-Mn/TiO₂ Catalysts for Selective Catalytic Reduction of NO with NH₃ at Low Temperature

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The sulfur tolerance of Ca modified MnO_x/TiO₂ catalysts in low-temperature SCR process was investigated. Experimental results revealed that the durability of developed catalysts in the presence of SO₂ could be improved by Ca modification. After being subjected to a range of analytical techniques, it was found that the surface Ca species could act as a SO₂ trap by preferentially reacting with SO₂ to form bulk-like CaSO₄, inhibiting the sulfation of active phase. Furthermore, the introduction of SO₂ had also preserved part of Lewis sites over the MnO_x. Both of these are conducive to NH₃ adsorption and activation at low temperature, hence improving the sulfur tolerance of Ca doped catalysts.

1. Introduction

Selective Catalytic Reduction (SCR) of NO_x with NH₃ in excess O₂ is one of the most effective ways to eliminate NO_x emissions from stationary sources [1]. However, by using commercialized V₂O₅/TiO₂-based catalysts, the SCR reactors have to be located upstream of the particulate control device due to their high operation temperature. This brings inherent problems like SO₂ oxidation, installation difficulty, and high deactivation risk. Therefore, the development of low-temperature SCR catalysts has attracted great research interests in recent years [2].

Up to now, lots of transitional metal modified low-temperature catalysts have been reported, among which, manganese oxides have been studied extensively due to their superior low-temperature SCR activity [3–9]. However, the Mn-based catalysts still suffer from SO₂ poisoning due to the presence of residual SO₂ (even after desulphurization), whose deactivation mechanism has been reported due to the sulfation of active phase or/and the blockage of catalysts' micropores by the deposition of (NH₄)₂SO₄ and NH₄HSO₄ [10]. As such, improvement of sulfur tolerance of the low-temperature SCR catalysts was also needed to widely concern.

One of the effective ways is the modifications by adding some metal oxides into the catalysts. Chang et al. [6] had reported that the addition of Sn into MnO_x-CeO₂ catalysts could greatly enhance the SO₂ resistance owing to the enhanced Lewis acid sites. Shen et al. [11] concluded that the iron doping would improve SO₂ tolerance of Mn-Ce/TiO₂ due to the inhibition of surface sulfate formation. Our previous work had demonstrated that adding Ce to Mn/TiO₂ could inhibit the active phase sulfation, contributing to the promotion in SO₂ resistance [12].

In our previous study [13], we reported that the Ca doping could greatly improve the low-temperature SCR activity of Mn/TiO₂ catalysts due to its positive effects on MnO_x dispersion and adsorptive capacity of NO_x, but the work did not look insightfully into the reaction behaviors of these catalysts in the presence of SO₂. As an alkali earth metal, Ca might have stronger chemical affinity to SO_x, which could lessen the sulfation of MnO_x, thereby enhancing the sulfur tolerance of catalysts. Similar finding was also observed by Du et al. [14] for Cu-Ce/TiO₂ system. Therefore, in this paper, the effects of Ca doping on SO₂ tolerance of Mn/TiO₂ catalysts were performed and the detailed mechanisms were analyzed by using XRD, BET, XPS, and DRIFT.

2. Experimental

2.1. Catalyst Preparation. The Ca-modified Mn/TiO₂ catalysts were prepared via a sol-gel method using butyl tetratitanate (0.1 mol), ethanol (0.8 mol), water (0.6 mol), acetic acid (0.3 mol), manganese nitrate (0.04 mol), and certain amount of calcium nitrate as we reported in the previous work [13]. The catalysts were hereafter denoted by Ca(*x*)Mn(0.4)/Ti, where *x*, 0.4 represented the molar ratio of CaO to TiO₂ and MnO_x to TiO₂, respectively.

2.2. Catalytic Activity Measurements. SCR activity measurements were carried out in a fixed-bed, quartz tubular flow reactor within the 60–200°C range of temperature. The typical reactant gas composition was NO of 600 ppm, NH₃ of 600 ppm, SO₂ of 50 ppm, O₂ of 3%, and balanced N₂, and the GHSV (gas hourly space velocity) was 40,000 h⁻¹. The concentrations of NO, NO₂, and N₂O were monitored by nondispersive infrared- (NDIR-) based gas analyzer (Photon-PGD-100 Madur Electronics).

2.3. Characterization. X-ray diffraction patterns (XRD) were recorded on a Rigaku D/Max-RA powder diffractometer using Cu Kα radiation (40 kV and 150 mV). X-ray photoelectron spectroscopy (XPS) was recorded with Al Kα X-rays (Thermal, ESCALAB 250). FT-IR spectra were acquired using an *in situ* DRIFT cell equipped with a gas flow system (Nicolet 6700 FTIR spectrometers). Samples were pretreated at 400°C in a He environment for 2 h and then cooled to 160°C. The background spectrum was recorded with flowing He and was subtracted from the catalyst spectrum.

3. Results and Discussion

3.1. Catalytic Activity in the Presence of SO₂. The effects of SO₂ on the SCR activities of Mn/TiO₂ and Ca doped Mn/TiO₂ catalysts were illustrated in Figure 1, which showed that SO₂ had obvious poisoning effect on SCR activity over all the catalysts. The Mn(0.4)/TiO₂ catalyst was much more susceptible to SO₂ than the Ca doped ones, where the NO conversion of Mn(0.4)/TiO₂ catalyst declined from 100% to 30% after introducing 50 ppm SO₂ over 4 h, but about 80% NO conversion was preserved over the Ca(0.1)-Mn(0.4)/TiO₂ catalyst. This indicated that the Ca doping could enhance the SO₂ tolerance of Mn/TiO₂ catalyst to some extent. However, the deactivation of catalysts could not be avoided in long-term running.

3.2. XRD and XPS Results

3.2.1. XRD Analysis. Figure 2 showed the XRD spectra of Mn(0.4)/TiO₂ and Ca(0.1)-Mn(0.4)/TiO₂ catalysts before and after SO₂ poisoning (fresh catalysts and used catalysts after SCR reaction in the presence of SO₂). After SO₂ poisoning, no obvious differences were observed on Mn(0.4)/TiO₂ catalyst, but peaks for CaSO₄ (PDF-no. 30-0279) were detected on Ca(0.1)-Mn(0.4)/TiO₂ catalysts, indicating the formation of bulk-like CaSO₄ during the SCR reaction. There

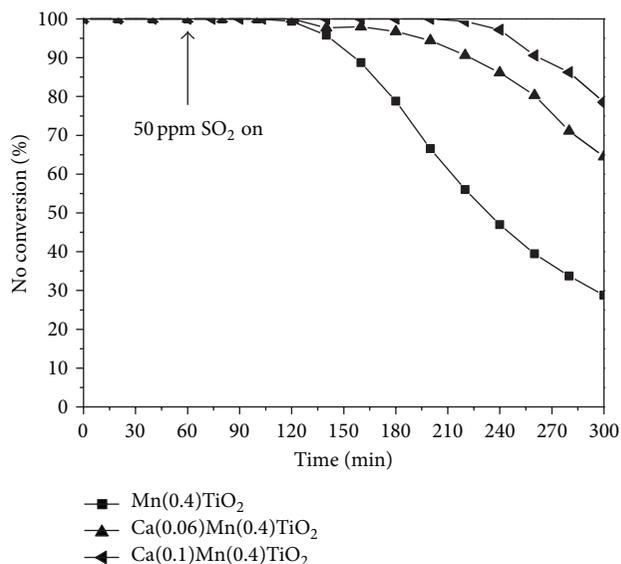


FIGURE 1: SCR activities of Mn/TiO₂ and Ca-Mn/TiO₂ prepared by a sol-gel method in the presence of SO₂ at 160°C ([NH₃]=[NO] = 600 ppm, [O₂] = 3%, [SO₂] = 50 ppm, N₂ balance, GHSV = 40,000 h⁻¹).

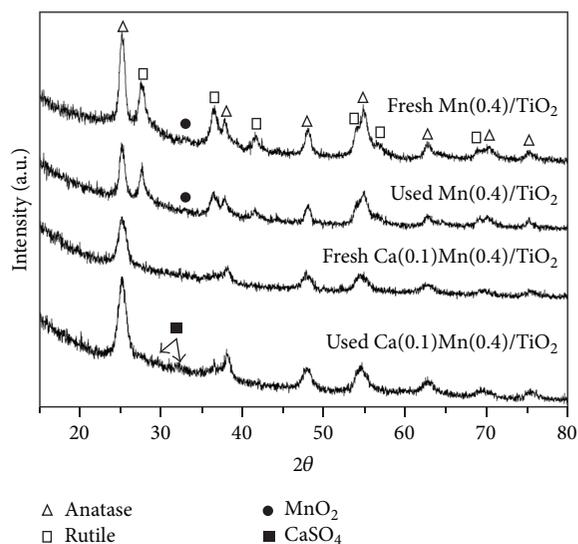


FIGURE 2: XRD patterns of Mn/TiO₂ and Ca(0.1)-Mn/TiO₂ before and after SCR reaction in the presence of SO₂: Δ, anatase; □, rutile; ●, MnO₂; ■, CaSO₄.

were no peaks for (NH₄)₂SO₄ and NH₄HSO₄ that existed for both catalysts, suggesting that the deactivation under SO₂ atmosphere could be due to the sulfation of active phase. The BET results (see Table S1) also confirmed this assumption as only minor changes of pore volume and surface area were detected.

3.2.2. XPS Analysis. The photoelectron spectra of Mn 2p for different catalysts before and after SO₂ poisoning were displayed in Figures 3(a) and 3(b), respectively. After being

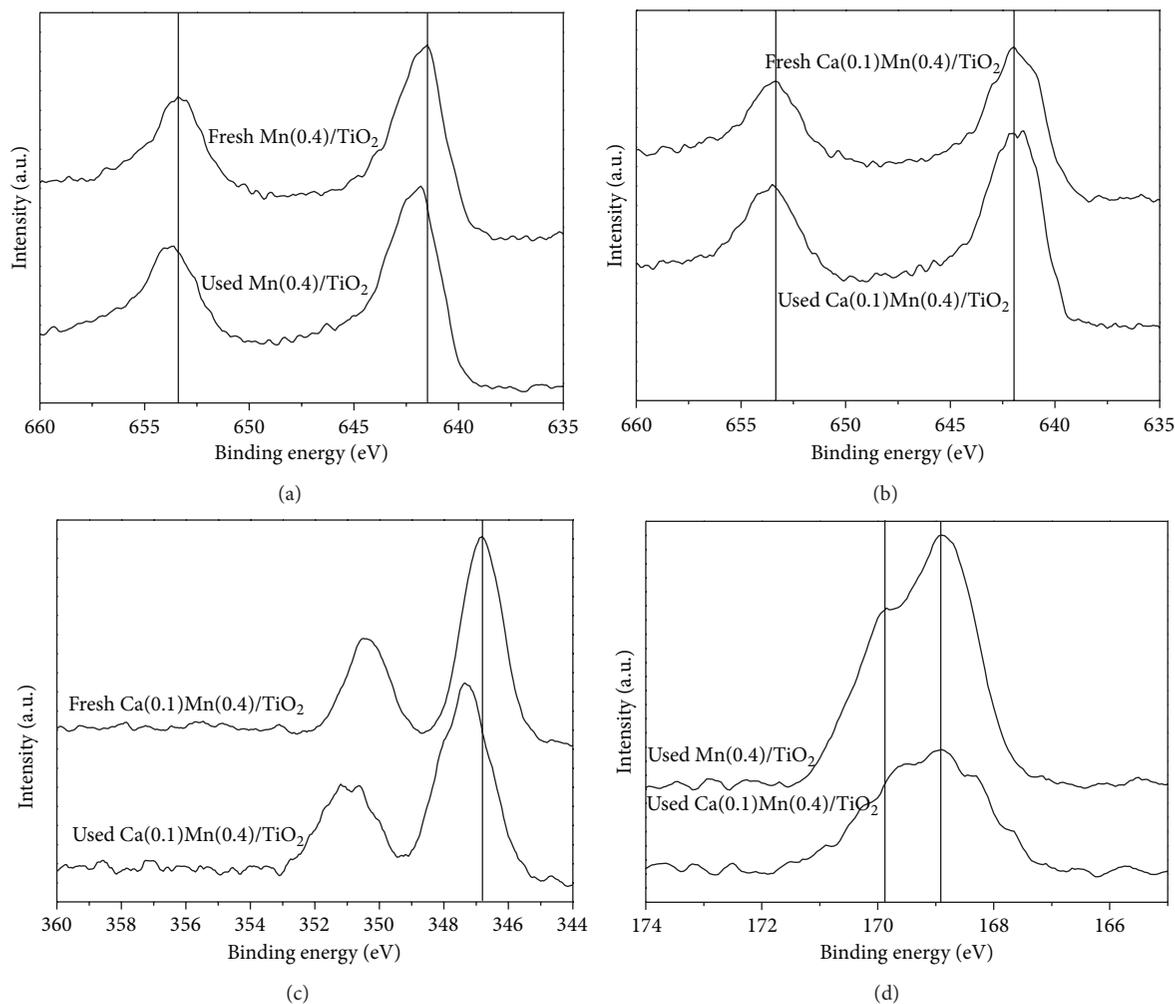


FIGURE 3: XPS spectra of Mn/TiO₂ and Ca(0.1)-Mn/TiO₂ before and after SCR reaction in the presence of SO₂ ((a) Mn 2p Mn/TiO₂, (b) Mn 2p Ca(0.1)-Mn/TiO₂, (c) Ca 2p, and (d) S 2p).

pretreated by SO₂, Mn 2p_{3/2} peak of Mn(0.4)/TiO₂ obviously shifted to about 0.5 eV higher binding energy, which indicated that some new surface species were formed. Similar phenomena were reported that the XPS peaks of metal-oxides catalysts could migrate to higher binding energy due to the active phase sulfation [15, 16]. Therefore, we could conclude that the MnO_x was sulfated during the SCR in the presence of 50 ppm SO₂. In contrast, for Ca doped Mn(0.4)/TiO₂ catalyst, there were no noticeable changes in the binding energy of Mn 2p_{3/2} being observed after SO₂ introduction, which illustrated that the Ca addition could efficiently restrain the sulfation of MnO_x on catalysts surface, shielding the active phases. Figure 3(c) revealed the binding energies of Ca 2p photoelectron peaks. For fresh Ca(0.1)-Mn(0.4)/TiO₂, the binding energy of Ca 2p_{3/2} was 346.8 eV, which was close to the value of CaO (347.2 eV) reported in the literature [17]. After SO₂ treated, the XPS peaks of Ca 2p_{3/2} shifted to higher binding energy range. Combined with the XRD results, the shift in binding energy was mainly ascribed to the formation of sulfated calcium (Ca 2p peaks for CaSO₄ at about 348.1 eV) [18]. Figure 4(d) also represented

the photoelectron spectra of S 2p for Mn/TiO₂ and Ca(0.01)-Mn/TiO₂ after SO₂ poisoning. The peaks at 168.9 and 169.8 eV all could be attributed to the S(VI), indicating the formation of sulfate species after SO₂ poisoning [19, 20].

Additionally, it can be also observed that the surface sulfur content of Ca(0.1)-Mn(0.4)/TiO₂ was lower than that of Mn(0.4)/TiO₂ after SCR reaction (see Table S2), which suggested that the deposition of sulfate species on catalyst surface was inhibited after Ca doping. The possible reason is that Ca dopants would preferentially react with SO₂ to form CaSO₄ and thereby weaken the sulfation of MnO_x. However, it should be noted that the sulfate species could also migrate into the bulk phase as CaSO₄ (see XRD results) would also lead to the decline of surface sulfur content.

3.3. In Situ DRIFT Study

3.3.1. SO₂ Adsorption on Mn/TiO₂ and Ca-Mn/TiO₂. Figure 4 showed the DRIFT spectra of adsorbed species over Mn(0.4)/TiO₂ and Ca(0.1)-Mn(0.4)/TiO₂ catalysts in

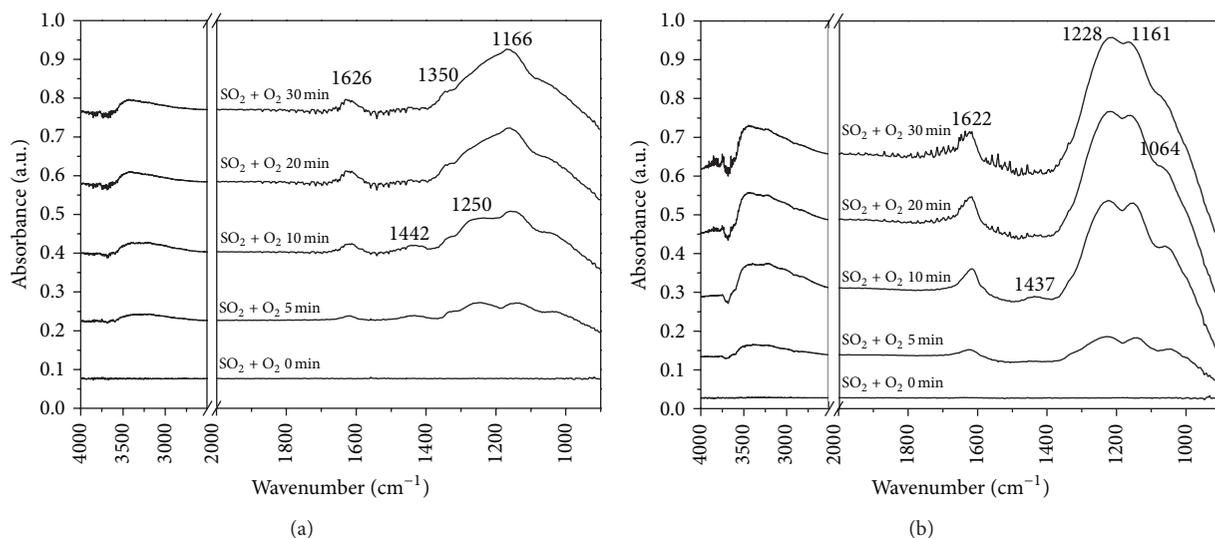


FIGURE 4: DRIFT spectra of Mn/TiO₂ and Ca(0.1)-Mn/TiO₂ exposed to SO₂ at 160°C.

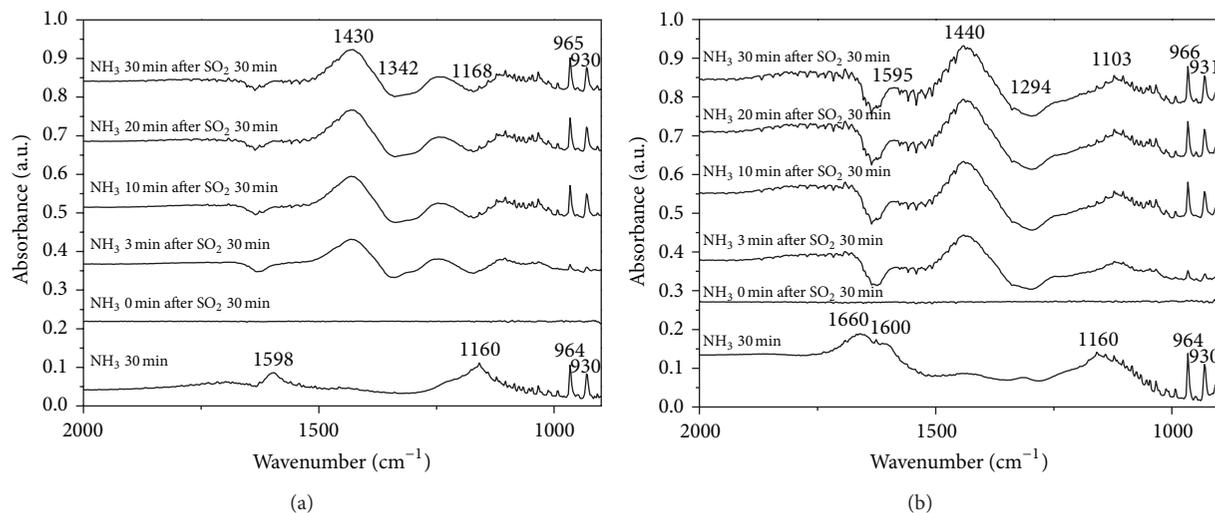


FIGURE 5: DRIFT spectra of Mn/TiO₂ and Ca(0.1)-Mn/TiO₂ exposed to NH₃ after SO₂ pretreated at 160°C.

flowing SO₂ + O₂/He at 160°C as a function of time. As shown in Figure 4(a), several bands at 1166, 1250, 1350, 1442, and 1626 cm⁻¹ were attributed to adsorbed sulfate species. The bands at around 1200 cm⁻¹ (1166 and 1250 cm⁻¹) were caused by bulk-like sulfated species and assigned to the vibrations of S–O and S=O [21–23], and the bands at 1442 and 1350 cm⁻¹ were caused by the bonds vibrations of surface sulfate [22]. Moreover, the bands around 1626 cm⁻¹ were assigned to the adsorbed H₂O due to the reaction between SO₂ and surface hydroxyl groups [24]. In contrast, the IR spectra of Ca(0.1)-Mn(0.4)/TiO₂ were somewhat different from that of Mn(0.4)/TiO₂ as shown in Figure 4(b), which is the characterized peaks of bulk sulfate [22]. According to the XRD and XPS results, these bulk sulfate species should be attributed to bulk-like CaSO₄. This indicated that after Ca

addition, the sulfate species tended to form in bulk rather than on surface, protecting the active phases of the catalysts.

3.3.2. NH₃ Adsorption after SO₂ Pretreatment on Catalysts' Surface. DRIFT spectra of NH₃ adsorption on Mn(0.4)/TiO₂ and Ca(0.1)-Mn(0.4)/TiO₂ catalysts at 160°C after SO₂ pretreatment for 30 min were presented in Figures 5(a) and 5(b), respectively. And as a comparison, the spectra of the fresh catalysts treated with NH₃ for 30 min were also shown in Figure 5 as the first curve. As shown in Figure 5(a), the spectrum taken after 30 min of NH₃ adsorption on Mn(0.4)/TiO₂ was characterized by bands at 930, 964, 1160, and 1598 cm⁻¹ [3]. The bands located in 930 and 964 cm⁻¹ were attributed to the weakly adsorbed NH₃ or gas phase NH₃. In the

NH stretching region, the bands at 1160, 1598 cm^{-1} were indicative of coordinative adsorbed NH_3 on Lewis sites [25–27]. As for SO_2 treated catalyst, new bands at 1430, 1342, and 1105 cm^{-1} emerged. According to the reports [8, 28], the band at 1430 cm^{-1} could be attributed to the bending vibrations of NH_4^+ ions formed on Brønsted acid sites and the band at 1105 cm^{-1} could be assigned to the hydrogen bonds of adsorbed NH_3 [21]. Compared with the fresh sample, SO_2 pretreatment would significantly promote the formation of Brønsted acid sites, where the Lewis acid sites almost disappeared after introducing SO_2 . Moreover, the negative peak at 1342 cm^{-1} was ascribed to S=O band, which tended to bond with NH_3 on catalyst surface according to the previous study [10].

As for Ca(0.1)-Mn(0.4)/ TiO_2 catalysts (Figure 5(b)), similar bands were observed for NH_3 adsorption after SO_2 pretreatment. The band at 1103 cm^{-1} was assigned to hydrogen-bonded NH_3 adspecies. The band at 1440 cm^{-1} was due to the NH_4^+ ions formed on Brønsted acid sites. The negative peaks at 1294 cm^{-1} could be attributed to (S=O) bond due to the interaction between sulfate species and NH_3 . However, the IR spectra also showed some differences with that for Ca-free catalyst. It was found that the band at 1598 cm^{-1} due to NH_3 adsorption on Lewis acid sites was preserved although the intensity was reduced, and the peak at 1160 cm^{-1} (NH_3 adsorption on Lewis acid sites) still can be detected, which was probably overlapped by the bands at 1103 cm^{-1} . Thus, since the NH_3 adsorption and activation on Lewis acid sites played a critical role in the low-temperature SCR process [25, 29], we can conclude that the lessen in MnO_x sulfation and the preservation of part of Lewis acid sites could be the key reason for the great enhancement in SO_2 tolerance of the catalysts after Ca addition.

4. Conclusion

Ca modifications of $\text{MnO}_x/\text{TiO}_2$ catalysts using a sol-gel method would bring an obvious enhancement in SO_2 tolerance for low-temperature SCR of NO with NH_3 . Experimental results showed that around 80% NO conversion could be remained for Ca(0.1)-Mn(0.4)/ TiO_2 catalyst in the presence of 50 ppm SO_2 for 4 h, while that was less than 30% for Ca-free catalyst. Based on the characterizations by XRD, XPS, and IR, it was concluded that the sulfation of MnO_x was greatly inhibited by Ca doping, which was assumed to be due to the fact that the Ca dopants would preferentially react with SO_2 to form bulk-like sulfate species. Furthermore, DRIFT results also indicated that part of Lewis acid sites could be preserved on Ca doped catalysts that was pretreated with SO_2 , which was beneficial to the fulfillment of the low-temperature SCR cycle.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

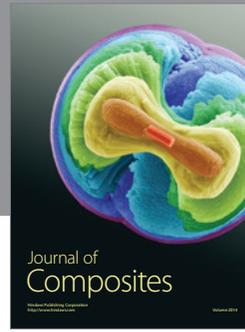
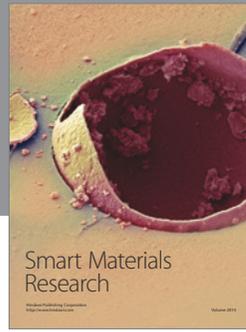
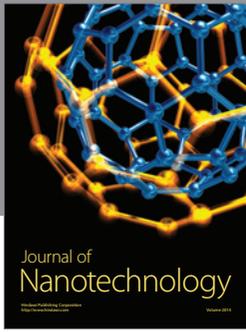
Acknowledgments

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