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Research Article

Promotional Effect on Selective Catalytic Reduction of NO_x with NH_3 over Overloaded W and Ce on V_2O_5/TiO_2 Catalysts

Seunghee Youn, Inhak Song, and Do Heui Kim

School of Chemical and Biological Engineering, Institute of Chemical Processes, Seoul National University, Seoul 151-744, Republic of Korea

Correspondence should be addressed to Do Heui Kim; dohkim@snu.ac.kr

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W and Ce are known to be a good promoters to improve selective catalytic reduction (SCR) activity for V_2O_5/TiO_2 catalysts. This work aimed at finding the optimum ratio and loading of promoters (W and Ce) on V_2O_5/TiO_2 catalyst in order to improve SCR reactivity in low temperature region and to minimize N_2O formation in high temperature region. In addition, we changed the order of impregnation between W and Ce precursors on V_2O_5/TiO_2 catalyst during the preparation and observed its effect on SCR activity and N_2 selectivity. We utilized various analytical techniques, such as N_2 adsorption-desorption, X-ray diffraction (XRD), and temperature-programmed reduction with hydrogen (H_2 TPR) to investigate the physicochemical properties of catalysts. It was found that W- and Ce-overloaded V_2O_5/TiO_2 catalyst such as W/Ce/V/TiO_2 (15 : 15 : 1 wt%) showed the most remarkable DeNO$_x$ properties over the wide temperature region. Additionally, this catalyst significantly suppressed N_2O formation during SCR reaction, especially in high temperature region (350–400°C). Based on the characterization results, it was found that such superior activity originated from the improved reducibility and morphology of W and Ce species on V_2O_5/TiO_2 catalyst when they are incorporated together at high loading.

1. Introduction

Selective catalytic reduction of NO_x with NH_3 (NH_3-SCR) as reductant is well-known technology for removing NO_x emissions from stationary source or diesel engine vehicles operated under highly oxidizing condition [1–5]. Generally, vanadia-titania-based NH_3-SCR catalysts have been used in stationary sources due to high resistance to SO_2, while Cu-based zeolite is being used for mobile source [6]. In spite of a high NO_x conversion between 300 and 400°C, the side reactions such as the oxidation of NH_3 to N_2O or NO and toxicity of the evaporated vanadium at high temperature are the major problems of V_2O_5/TiO_2 catalysts to overcome [7, 8].

Meanwhile, International Maritime Organization (IMO) announced the NO_x regulations (Tier III) on vessels, which will take effect from 2016 [9]. It makes catalyst community pay attention to the DeNO$_x$ technology for marine application. In particular, the SCR catalyst for marine application requires higher DeNO$_x$ activity in the low temperature range (200–250°C) due to the low temperature exhaust from marine engine. In addition to needs to lower the SCR operation temperature, a concern for the emissions of N_2O has been growing, since N_2O has about 300 times greater greenhouse gas effect than CO_2. During NH_3 SCR reaction, N_2O is known to be produced in the following reaction:

$$\begin{aligned} 4NO + 4NH_3 + 3O_2 &\longrightarrow 4N_2O + 6H_2O \\ 2NH_3 + 2O_2 &\longrightarrow N_2O + 3H_2O \end{aligned} \tag{1}$$

In commercial catalysts, the loading of vanadia is generally as low as 1–1.5 wt% whereas the loading of promoters is about 6–10 wt%, respectively [3]. W-V/TiO_2 or Ce-V/TiO_2 is well-known SCR catalytic system for commercial application. However, there is no previous study regarding the overloading effect (higher than 10 wt%) of promoters on V_2O_5/TiO_2 SCR catalysts to our knowledge. Therefore, the aim of this study is to find the optimum condition to prepare V_2O_5/TiO_2 SCR catalyst with high DeNO$_x$ activity in low temperature and minimized N_2O formation by loading the excessive amount of promoters (W and Ce) compared with vanadium loading. Additionally, we tried to change the order of impregnation between two promoters on V_2O_5/TiO_2 while focusing on the characteristics of catalysts. We also investigate whether the synergetic effect of promoters exists when they are loaded together on V_2O_5/TiO_2. To sum up, the effect of impregnation order and the ratio between promoters and vanadium in $M/V_2O_5/TiO_2$ catalyst on the NO_x conversion and the N_2O selectivity during SCR reaction were addressed in this study.

2. Experimental

2.1. Catalysts Preparation. All catalysts were prepared by applying wet impregnation method of vanadium and promoter solution on anatase titania. Anatase TiO_2 powder (DT-51 Millennium Chemicals) was used as support. Ammonium metavanadate (99%, Sigma Aldrich) was dissolved in diluted oxalic acid solution (0.5 M) to produce the solution of vanadium precursor [10]. The samples with 1 wt%, 3 wt%, and 5 wt% V_2O_5 loading on TiO_2 were prepared. We used two promoters, W and Ce, to be impregnated on V_2O_5/TiO_2 catalyst. Ammonium tungstate (99.99%, Sigma Aldrich) and cerium(III) nitrate hexahydrate (99.99%, Sigma Aldrich) were used to produce precursor solution which was dissolved in diluted water. After drying at 105°C for 12 h, promoter precursor solution was added to V_2O_5/TiO_2 in a rotary evaporator again. After a series of impregnation processes, catalysts were dried and then calcined at 400°C for 4 h in air.

Sample was designated as the following notation M1/M2//VT ($X:Y:Z$). M1 and M2 showed the promoters and sequence of impregnation, where M2 is firstly impregnated on V_2O_5/TiO_2, followed by the impregnation of M1 on $M2/V_2O_5/TiO_2$. X and Y meant the weight loading of each promoter and Z is the weight loading of vanadium on TiO_2. For example, W/Ce/VT (15 : 15 : 1) indicated V_2O_5/TiO_2 catalyst with 1 wt% of vanadium loading which are promoted with W and Ce promoters with 15 wt% each.

2.2. Characterization. The specific surface area of catalysts was measured by ASAP 2010 (Micromeritics) apparatus and calculated by using BET method. The samples about 0.1 g were degassed at 250°C for 12 h before measurement. After the pretreatment step, N_2 adsorption-desorption isotherms were obtained at −196°C. The X-ray diffraction (XRD) patterns were obtained by using a Rigaku mode 1 Smartlab with Cu Kα radiation ($\lambda = 0.1542$ nm). 40 kV and 50 mA of voltage and current were applied and the XRD patterns were collected in a 2-theta range from 20 to 80° at a speed of 10°/min [11].

H_2 TPR was used to measure the reducibility of metal oxides by detecting the hydrogen consumption at elevated temperature with a thermal conductivity detector (TCD) in a BEL-CAT-BASIC (BEL Japan Inc.). Prior to the analysis, 0.03 g samples were pretreated at 350°C for 1 h in a flow of 30 mL/min Ar. After cooling down to room temperature, samples were then exposed to 5% H_2/Ar until the temperature reached 800°C at the rate of 10°C/min.

2.3. Reaction Test. SCR activity was measured in a fixed-bed quartz tubular reactor. Catalysts were sieved to 300–500 μm in diameter then loaded in the reactor. The composition of inlet gas was 500 ppm NO, 500 ppm NH_3, and 2% O_2 and was balanced with N_2. Space velocity of inlet gas was maintained to be 40,000 h^{-1}. We raised reaction temperature from 150°C to 400°C in increments of 50°C with the rate of 10°C/min. NO_x concentration of outlet gas was analyzed by using NO_x chemiluminescence analyzer (Model 42i High level, Thermo Scientific). Also, Fourier transform infrared (FT-IR) spectroscopy was applied to observe the N_2O concentration in the gas. We used the average data of 16 scans at a resolution of 1.0 cm^{-1}. A Nicolet 6700 (Thermo Scientific), with 2 m gas analysis cell heated to 120°C to exclude the effect of H_2O, was used for gas phase analysis. N_2 selectivity referred to in this study was calculated by using the following equation [2]:

$$N_2 \text{ selectivity} (\%) = \frac{[[NO]_{in} + [NH_3]_{in}] - [[NO]_{out} - 2[N_2O]_{out}]}{[[NO]_{in} + [NH_3]_{in}]} \times 100\%. \quad (2)$$

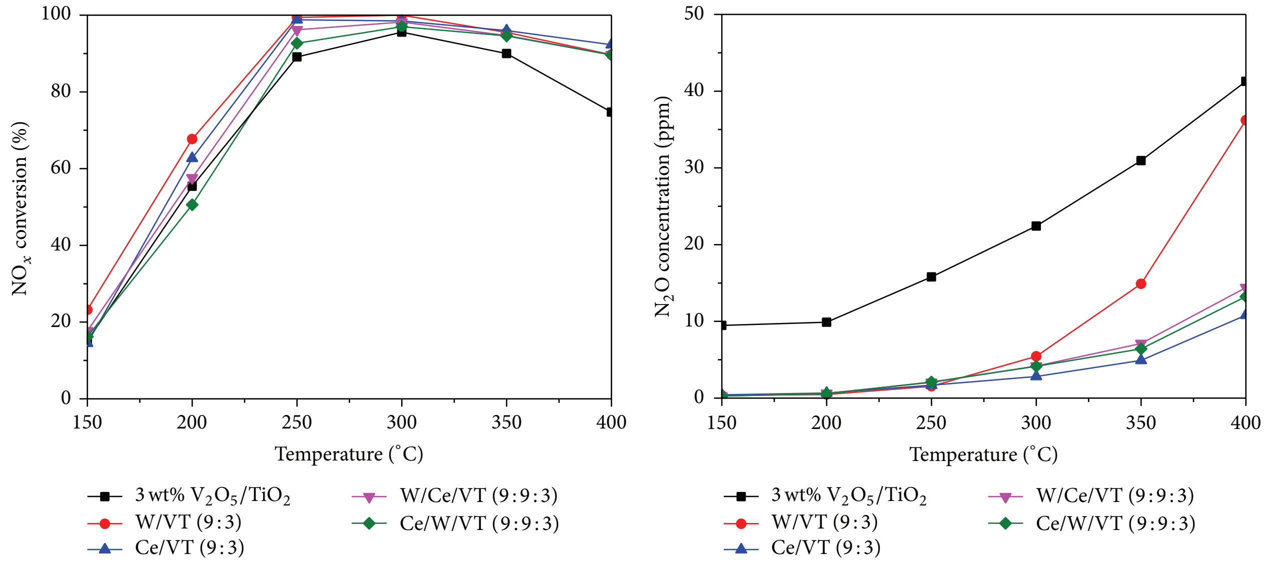
Table 1: NO_x conversion at 200°C and N_2O concentration at 400°C obtained on all the samples we tested in this study.

Sample	NO_x conversion (%) at 200°C	N_2O concentration (ppm) at 400°C
1 wt% V_2O_5/TiO_2	10	15.4
3 wt% V_2O_5/TiO_2	55	41.3
5 wt% V_2O_5/TiO_2	67	42.1
W/VT (9 : 3)$^{(a)}$	67	36.2
Ce/VT (9 : 3)	63	10.8
W/Ce/VT (9 : 9 : 3)	58	14.4
Ce/W/VT (9 : 9 : 3)	51	13.2
W/VT (9 : 1)	34	6.9
Ce/VT (9 : 1)	41	5.6
W/Ce/VT (9 : 9 : 1)	36	5.0
Ce/W/VT (9 : 9 : 1)	33	5.2
W/VT (15 : 1)	28	6.7
Ce/VT (15 : 1)	44	3.7
W/Ce/VT (15 : 15 : 1)$^{(b)}$	68	4.2
Ce/W/VT (15 : 15 : 1)$^{(c)}$	55	3.7

(a), (b), and (c) were chosen as the promising catalysts.

3. Result and Discussion

Two representative elements, W and Ce, were chosen as promoters to V_2O_5/TiO_2. The order of impregnation and loading was changed to find the optimum catalyst. All results of prepared catalysts were displayed in Table 1. Since high SCR activity at low temperature and low N_2O formation at high temperature are regarded as two main requirements for novel SCR catalysts, we chose NO_x conversion at 200°C and N_2O concentration at 400°C to compare the samples we tested in this study. Firstly, Figure 1 showed NO_x conversion and N_2O concentration of various samples containing 3 wt%



(a)

(b)

Figure 1: (a) NO$_x$ conversion and (b) N$_2$O concentration of various samples containing 3 wt% vanadium and 9 wt% promoters.

V_2O_5 with different amounts of W and Ce. Each SCR reaction result was compared with standard SCR catalysts with same vanadium loading.

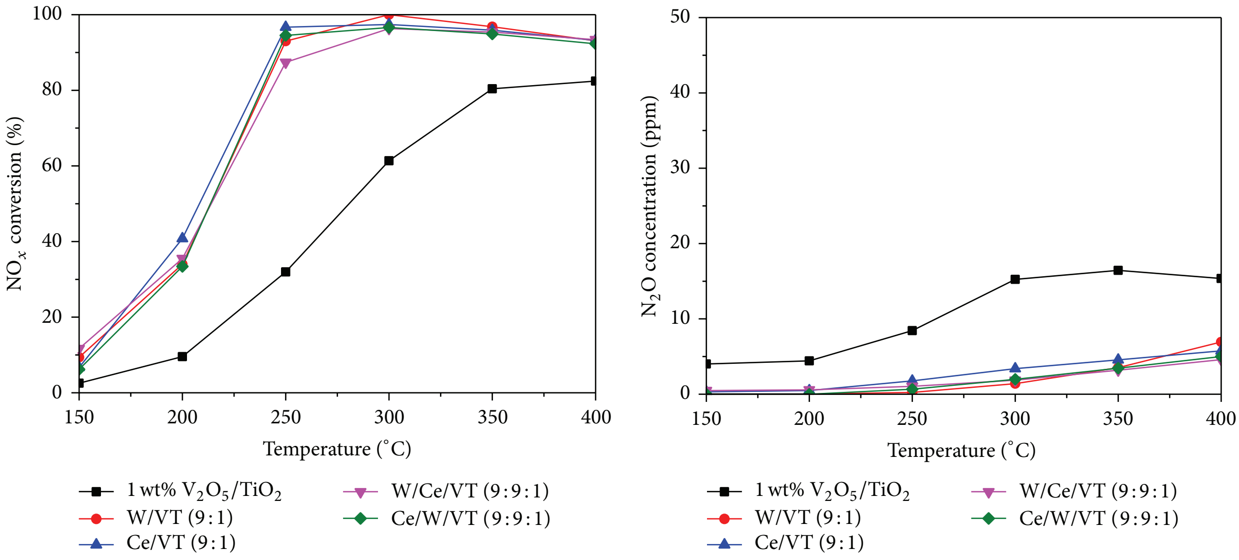
As shown in Figure 1, the addition of 9 wt% promoters on 3 wt% V_2O_5/TiO_2 (W/VT (9 : 3) or Ce/VT (9 : 3)) demonstrated the slightly higher NO$_x$ conversion than 3 wt% V_2O_5/TiO_2 catalysts not only in the low temperature region (250°C) but also in the high temperature region (400°C). However, W/Ce/VT (9 : 9 : 3) or Ce/W/VT (9 : 9 : 3) catalyst, which were expected to show synergetic effect of two promoters, showed similar SCR activity in low temperature region below 250°C to standard sample, implying that no synergism between two promoters exists at this condition. It must be pointed out that all W- and Ce-containing catalysts displayed lower N_2O formation at 400°C than 3 wt% V_2O_5/TiO_2. 3 wt% V_2O_5/TiO_2 catalyst showed 81% N_2 selectivity whereas Ce-containing catalysts showed about 91%.

The ratio of promoter to vanadium was increased by lowering the vanadium loading to 1 wt% to see whether the promotional effect is enhanced. As shown in Figure 2, all the promoter-containing catalysts demonstrated considerably improved NO$_x$ conversion, especially in low temperature range, compared with 1 wt% V_2O_5/TiO_2, implying that the promotional effect is remarkably enhanced over the catalyst with the low vanadium loading (1 wt%). As observed in the Figure 1, however, the addition of two promoters, for example, W/Ce/VT (9 : 9 : 3), did not give rise to the enhanced NO$_x$ conversion over all temperature ranges.

Since we observed the promotional effect of adding promoters (9 wt% Ce or W) on SCR activity, we further increased the amount of promoters to 15 wt%. As shown in Figure 3, the increase in W loading from 9 wt% to 15 wt% resulted in the rather decrease in the activity at 200°C, while that of Ce loading showed almost the same activity. More drastic enhancement of activity in the low temperature region was observed for the samples such as W/Ce/VT (15 : 15 : 1) or Ce/W/VT (15 : 15 : 1). For example, W/Ce/VT (15 : 15 : 1) catalyst displayed 68% of NO$_x$ conversion at 200°C which was the best activity among all catalysts. This value was as high as that of W/VT (9 : 3) catalyst; however, more important thing to note would be even lower N_2O production (30 ppm versus 5 ppm) at 400°C.

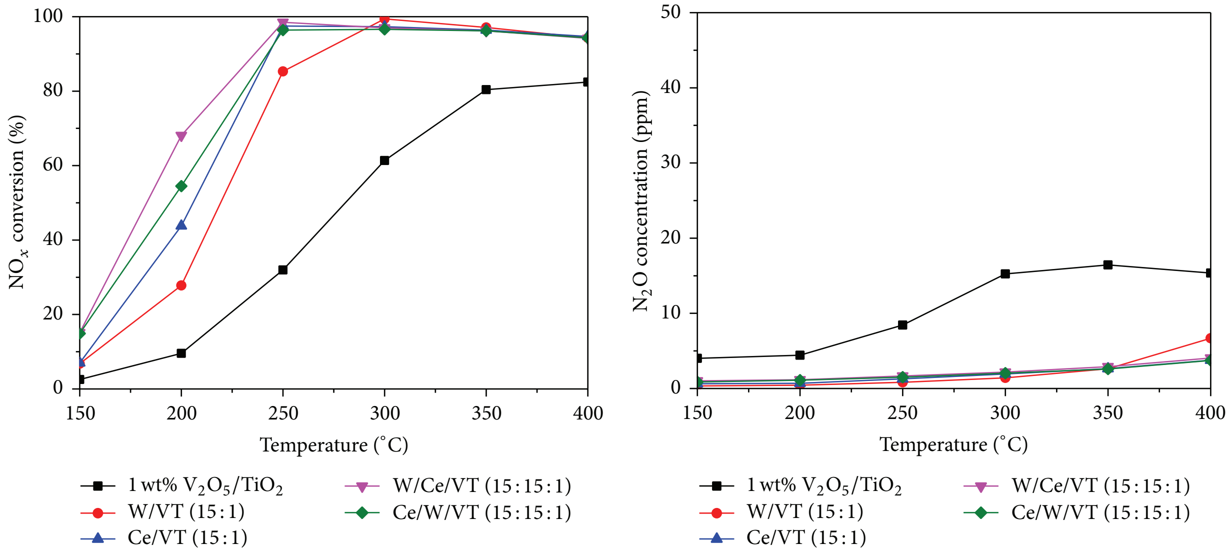
Noteworthy is that the synergism between Ce and W elements is observed only at higher ratio of promoters to vanadium. For the case of W/Ce/VT (9 : 9 : 1) catalyst, there is no promotional effect upon simultaneous loading of two promoters. On the other hand, we observed confident synergistic effect over W/Ce/VT (15 : 15 : 1) sample, especially the NO$_x$ conversion in the low temperature region, although there is not much difference in N_2O production. It must be pointed out that, for most cases, the change of the impregnation order of W and Ce did not reveal remarkable effect on NO$_x$ conversion and N_2O formation during SCR reaction. According to the references, WO_3/TiO_2 [12] and CeO_2/TiO_2 [13] catalysts showed low emission of N_2O at high temperature, although the reason of low N_2O formation was not fully identified. Generally, vanadium-containing catalysts usually emit more N_2O than vanadium-free catalysts while the former has higher deNO$_x$ activity than the latter. Therefore, the result of reduced N_2O formation on vanadium-containing catalysts the same level as nonvanadium metal oxide catalysts on TiO_2 was a remarkable accomplishment of our study.

Figure 4 displayed how overloaded W- and Ce-containing catalysts improve SCR activity compared with pure V_2O_5/TiO_2 catalysts. W/Ce/VT (15 : 15 : 1) and Ce/W/VT (15 : 15 : 1) showed slightly higher NO$_x$ conversion than 5 wt% V_2O_5/TiO_2 catalyst over all temperature ranges; more importantly, they produced about 88% less N_2O at 400°C. It is worth



(a) (b)

FIGURE 2: (a) NO$_x$ conversion and (b) N_2O concentration of various samples containing 1 wt% vanadium and 9 wt% promoters.



(a) (b)

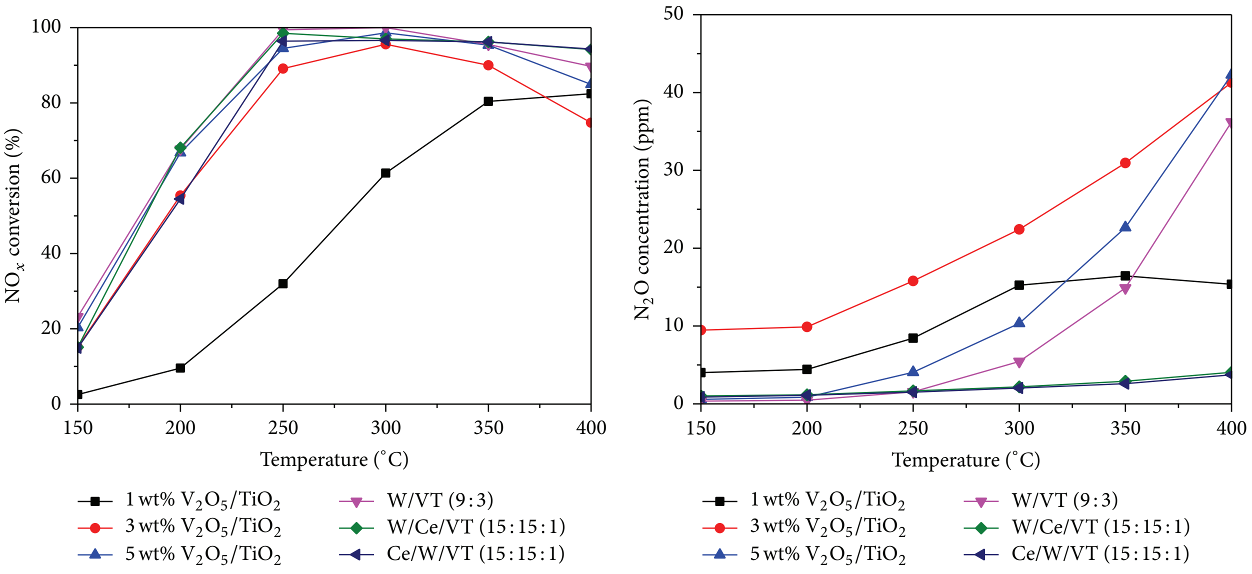
FIGURE 3: (a) NO$_x$ conversion and (b) N_2O concentration of various samples containing 1 wt% vanadium and 15 wt% promoters.

to mention that the higher activity and excellent N_2 selectivity can be obtained at low vanadium loading, which is regarded as the toxic element.

We applied various analytic techniques to investigate which characteristics made the difference in SCR reaction among variety of samples with different amount of promoters. Firstly, BET surface area of catalysts was measured by using N_2 adsorption-desorption method. Even samples containing overloaded Ce and W, in other words, Ce/W/VT (15 : 15 : 1) and W/Ce/VT (15 : 15 : 1) catalysts, showed the BET surface area of 60 m^2/g and 66 m^2/g, respectively. Since pure TiO_2 has the surface area of 87 m^2/g, it is confirmed that the overloading of W and Ce does not change the textural property of the catalyst too much, for example, by the blockage of pore.

As presented in Figure 5(a), XRD patterns were obtained to investigate the crystallinity of various catalysts containing 3 wt% of vanadium. Only anatase TiO_2 phase was observed in these catalysts with the peaks at 2 theta equal to 26°, 36°, 37°, 48°, 54°, 55°, 63°, 69°, 70°, and 75° [14, 15], except for the Ce/VT (9 : 3) sample in which very small peak at 28.5° arising from CeO_2 phase was brought out. This means that most of W or Ce promoters exist in the amorphous form or in a highly dispersed state on TiO_2 support.

When the vanadium loading decreased to 1 wt%, all catalysts also showed only TiO_2 main peaks, except for Ce/W/VT (9 : 9 : 1) catalyst displaying small CeO_2 peaks at 2 theta equal to 28°, 34°, 47°, and 56°, as demonstrated in Figure 5(b). However, the sample with the reversed impregnation order,



(a)

(b)

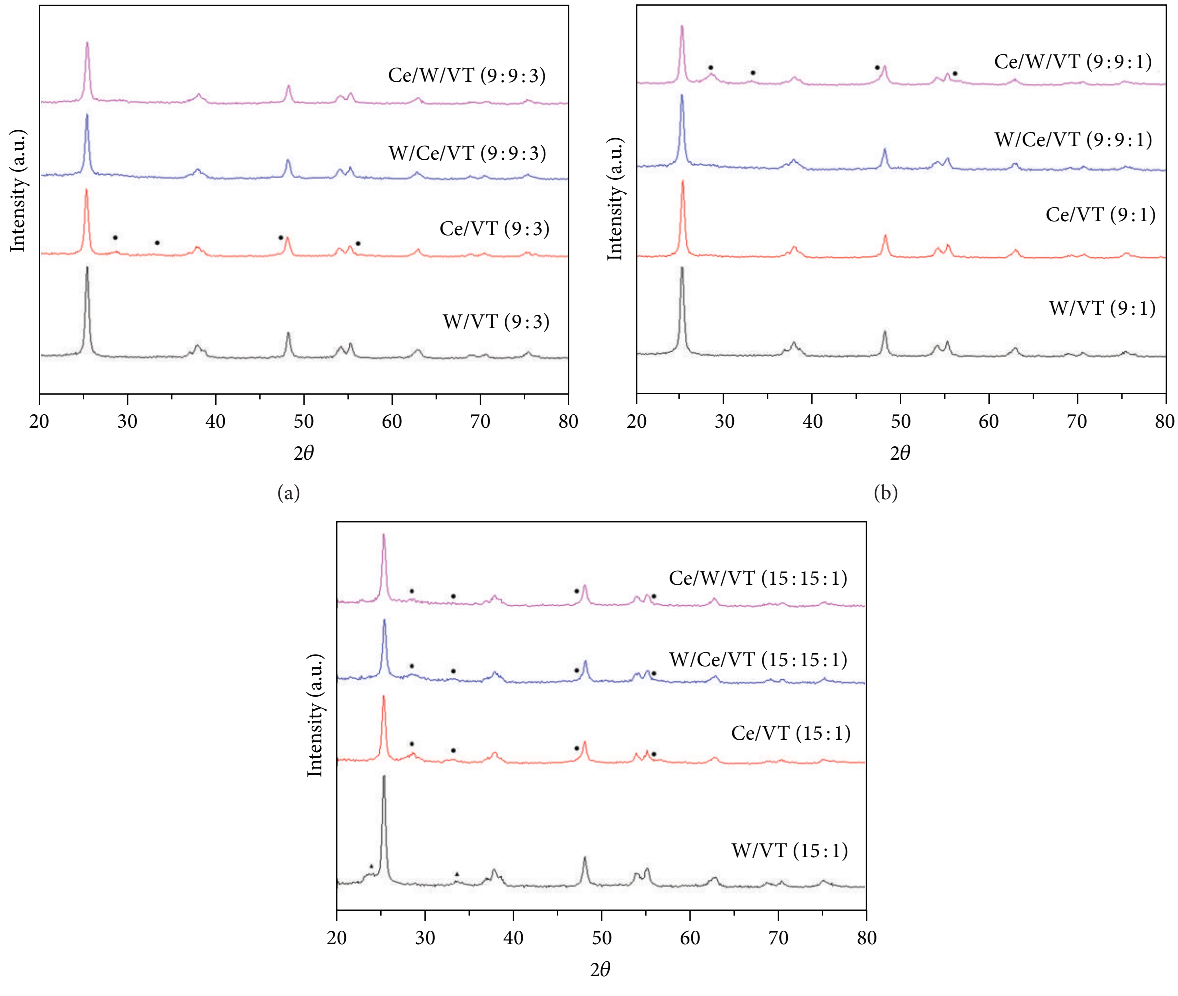
FIGURE 4: NO_x conversion and N_2O concentration of promising catalysts compared with 1 wt%, 3 wt%, and 5 wt% V_2O_5/TiO_2.

in other words, W/Ce/VT (9 : 9 : 1) catalyst, does not show any peaks other than TiO_2, implying that the impregnation order affects the crystallinity of the sample. As presented in Figure 5(c), all samples displayed small peaks in XRD patterns arising from either CeO_2 or WO_3. Specifically, broad CeO_2 peaks were observed in all 15 wt% Ce-containing catalysts. The samples containing both 15 wt% W and 15 wt% Ce, in other words, Ce/W/VT (15 : 1) and W/Ce/VT (15 : 15 : 1) catalysts, had lower crystalline CeO_2 phase compared with Ce/VT (15 : 1), implying that W aided in dispersing CeO_2 in the catalyst. In addition, crystalline WO_3 phase [16] was observed in the XRD pattern of W/VT (15 : 1) catalyst, which might have a negative effect on the activity as shown in Figure 3. However, the WO_3 phase was not detected for the samples such as W/Ce/VT (15 : 15 : 1) and Ce/W/VT (15 : 15 : 1) catalysts, where both Ce and W promoters were incorporated. Summarizing XRD patterns and activity measurement, it can be clearly confirmed that the synergistic effect observed in the samples such as W/Ce/VT (15 : 15 : 1) and Ce/W/VT (15 : 15 : 1) catalysts originated from the formation of highly dispersed Ce or W elements in the catalysts.

H_2 TPR is a widely used technique to investigate the redox properties of the catalysts. The H_2 TPR profiles of the prepared catalysts are shown in Figure 6. For the case of W/VT (15 : 1) sample, two H_2 consumption peaks at 500°C and 750°C were shown, which were assigned to the reduction of vanadium oxide and tungsten oxide, respectively [17]. On the other hand, Ce/VT (15 : 1) sample had the peak at 475°C, arising from the reduction of Ce^{4+} to Ce^{3+} reaction. The reduction behavior of the elements has changed when two promoters were incorporated together (W/Ce/VT or Ce/W/VT). It was reported that the introduction of Ce not only improved the oxygen storage capacity but also enhanced the redox activity by interaction between W and Ce species. The improvement of redox activity is confirmed by comparing the peak area of each sample. The peak area increased in the following order: W/VT (15 : 1) < Ce/VT (15 : 1) < W/Ce/VT (15 : 15 : 1) < Ce/W/VT (15 : 15 : 1). It is noticeable that two-promoter-containing catalysts showed larger peak area than only one promoter impregnated on V_2O_5/TiO_2. Samples which have larger peak area had more reducible form of metal oxide on their surfaces assisting higher NO_x reduction capability. Furthermore, the shift of the reduction peak of W/Ce/VT (15 : 15 : 1) and Ce/W/VT (15 : 15 : 1) compared with W/VT and Ce/VT indicated the close interaction between Ce and W species, resulting in the different Ce or W species on the surface. In addition, the fact that W/Ce/VT (15 : 15 : 1) showed higher NO_x conversion than Ce/W/VT (15 : 15 : 1) in low temperature region can be explained by the difference in reduction behavior of catalyst. Therefore, the simultaneous introduction of W and Ce could create a new center for oxygen storage and release, which can act as an active site for SCR reaction.

4. Conclusion

The promotional effect of loading W and Ce on V_2O_5/TiO_2 catalyst was investigated while changing different loading ratio between promoters and vanadium and the order of loading promoters in order to find the optimum catalyst for SCR reaction. Among the catalysts, W/Ce/VT (15 : 15 : 1) catalyst demonstrated the most desirable reaction behavior with high NO_x conversion and less N_2O formation during SCR reaction. It was found that highly dispersed CeO_2 and WO_3 phases were present even at high loading of W and Ce. Combined XRD and H_2 TPR results confirmed that the simultaneous impregnation of W and Ce on V_2O_5/TiO_2 at high loading leads to the change in the reducibility and



(a) (b) (c)

Figure 5: XRD patterns of catalysts with a ratio of promoter : vanadium—(a) 9 : 3, (b) 9 : 1, and (c) 15 : 1 (•: CeO_2, ▲: WO_3).

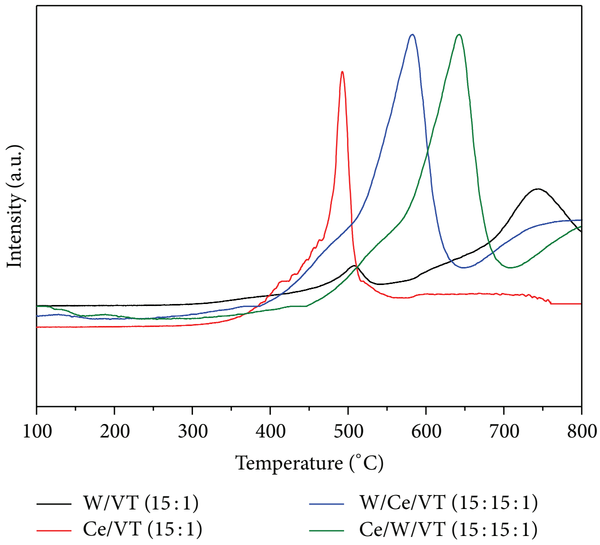


Figure 6: H_2 TPR profile of various samples containing 1 wt% vanadium and promoters.

morphology of elements in catalysts that eventually facilitated SCR reaction in low temperature region and minimized the amount of N_2O formed in high temperature region.

Conflict of Interests

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

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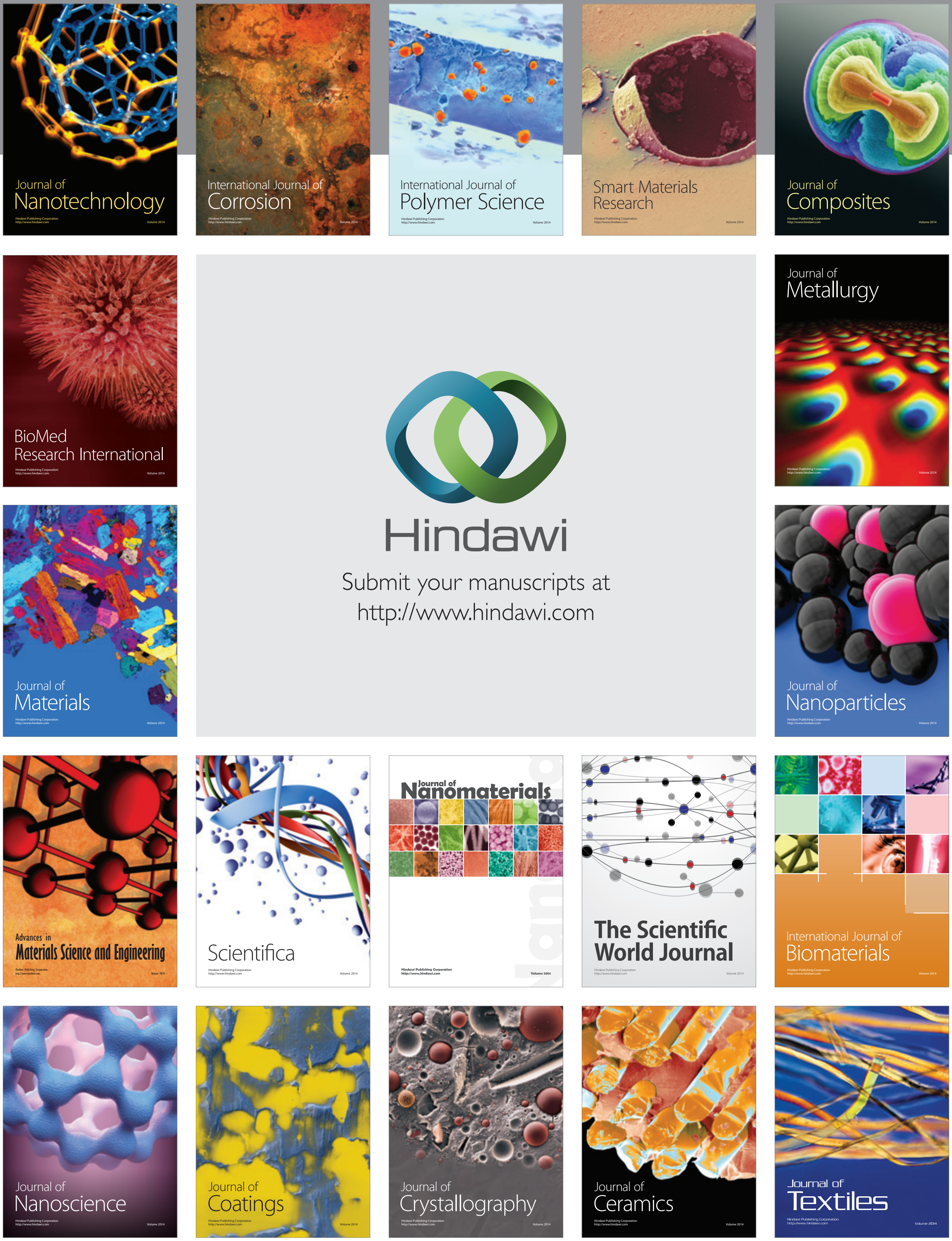

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