

Promoted Soot Oxidation by Doped $K_2Ti_2O_5$ Catalysts and NO Oxidation Catalysts

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ABSTRACT: In the present work, the promotion effects by doping metals on $K_2Ti_2O_5$ or by combining an additional NO oxidation catalyst are studied in detail for soot oxidation. The synthesized catalysts are characterized by X-ray diffraction. The promoting effects of NO and NO_2 on soot oxidation were examined on both $K_2Ti_2O_5$ and metal-doped $K_2Ti_2O_5$ catalysts. With pure $K_2Ti_2O_5$ catalyst, NO has a negligible promotion effect, because of the fact that $K_2Ti_2O_5$, different from Pt catalysts, is unable to oxidize NO to NO_2 . Compared to the pure $K_2Ti_2O_5$ catalyst, all the doped metals somehow enhance soot oxidation by decreasing the peak temperature (T_p). The activity follows the order of Pt/ $K_2Ti_2O_5$ (324 °C) > Co/ $K_2Ti_2O_5$ (331 °C) = Cu/ $K_2Ti_2O_5$ (331 °C) > Ce/ $K_2Ti_2O_5$ (335 °C) > $K_2Ti_2O_5$ (352 °C). With NO_2 , soot starts to be oxidized at temperatures as low as 250 °C, even with pure $K_2Ti_2O_5$. When $K_2Ti_2O_5$ is combined with a NO oxidation catalyst (Co/ $K_xTi_2O_5$), both the starting temperature (T_s) and T_p were greatly decreased, from 295 °C to 240 °C and from 352 °C to 326 °C, respectively. Therefore, the combination of $K_2Ti_2O_5$ with a NO oxidation catalyst can enhance the soot oxidation activity significantly.

1. INTRODUCTION

Because of their high efficiency, reliability, and durability, the sales of diesel-engine cars have been increasing significantly since the mid-1990s.^{1,2} The main pollutants emitted from diesel engines are nitrogen oxides (NO_x) and particulate matter (PM). Unfortunately, the PM in the nanoscale, which is classified as a carcinogen, can penetrate into the lung and cause severe bronchial troubles.³ As a result, the legislation levels of the emitted pollutants from diesel engines are becoming more and more stringent.⁴ However, these emission standards cannot be met anymore by only modifying the diesel engines and fuel additives. Thus, the development of after-treatment technologies to treat the diesel emissions is essential.^{5,6}

The most promising technique for the PM abatement is the diesel particulate filter trap (DPF).⁷ The PM compounds are first trapped on the filter and then are oxidized by O_2 or NO_2 . In this system, some catalysts are highly required to reduce the soot ignition temperature.⁸ The critical issue of the DPF is the development of an effective and economical catalyst. Currently, most of the oxidation catalysts used in the international markets are noble-metal-based materials. Because of the high cost of noble metals, many attempts have been made to develop noble-metal-free catalysts.^{9,10} During the last decades, several types of noble-metal-free catalysts have been developed, including perovskite-type oxides (such as $La_{1-x}K_xMO_3$ ($M = Fe, Co, Cu, \text{ or } Mn$)), spinel type oxides (such as $Cu_{1-x}K_xFe_2O_4$), alkaline or heavy-metal oxides (such as $CuO, V_2O_5, \text{ and } K/CeO_2$), and the mixtures of halides with vanadates or molybdates (such as $Cs_2O \cdot V_2O_5$).^{11–21} In addition to the catalytic activity, several other characteristics such as thermal stability and sulfur poisoning have also been taken into account. Normally, the temperature of the emitted gases could be very high. The majority of the

catalysts used in the real vehicles cannot survive the operating conditions and become sintered. In addition, the sulfur in the emitted gases can cause a permanent loss in activity by reacting with the active components.²²

It is widely accepted that potassium is the most active catalyst for soot oxidation, based on which many K-doped catalysts with high soot oxidation activities have been developed. However, the high mobility and low evaporation temperature of potassium limit their applications.^{23–26} To obtain an active soot oxidation catalyst with superior thermal stability, potassium-containing compounds can be one of the solutions.^{27,28} Okubo et al.²⁸ have reported that potassium-enriched nepheline group materials exhibit excellent soot oxidation activity and have claimed that the highly improved oxidation activity is attributed to the K species that exist on the surface of nepheline group materials. Recently, we discovered that soot combustion could be catalyzed by several potassium titanate compounds including $K_2Ti_2O_5$, $K_2Ti_4O_9$, and $K_2Ti_6O_{13}$, which were prepared by solid-state reaction between K_2CO_3 and TiO_2 at temperatures of 850–1100 °C. $K_2Ti_2O_5$ is observed to possess the highest activity for soot combustion.^{29,30} It is well-known that platinum, cobalt, cerium, and copper are active catalysts for soot oxidation. Therefore, in order to further increase the soot oxidation activity of $K_2Ti_2O_5$, some metal catalysts including platinum, copper, cobalt, or cerium were doped on $K_2Ti_2O_5$. The synergetic effects between $K_2Ti_2O_5$ and the supported metals for soot oxidation activities have not been reported yet.

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In the present work, the promotion effect of the doping metals such as copper, platinum, cerium, and cobalt on $K_2Ti_2O_5$ for soot combustion was investigated. Besides the direct promoted soot combustion by the doping metals, the indirect promotion by oxidizing NO to NO_2 by combining with one more NO oxidation catalyst, $Co/K_xTi_2O_5$, was also investigated.

2. EXPERIMENTAL SECTION

2.1. Catalyst Preparation and Characterization. $K_2Ti_2O_5$ was prepared from a stoichiometric amount of K_2CO_3 and TiO_2 precursors at 850 °C using a solid-state reaction method.³¹ Briefly, 6.91 g of K_2CO_3 (Yakuri Pure Chemical Co., Ltd.) was mixed with 7.99 g of TiO_2 (Hombikat UV 100) by ball milling for 24 h, followed by calcination at 850 °C for 10 h in air to obtain $K_2Ti_2O_5$.

$K_2Ti_2O_5$ catalysts doped with metals such as copper, platinum, cerium, or cobalt (denoted as $M/K_2Ti_2O_5$) are prepared following the same incipient wetness impregnation process.³² $Cu(NO_3)_2 \cdot 3H_2O$, $H_2PtCl_6 \cdot 5.7H_2O$, $Ce(NO_3)_3 \cdot 6H_2O$, and $Co(NO_3)_2 \cdot 6H_2O$ are used as metal precursors. Copper, cerium, or cobalt (5 wt % for each), and 2 wt % Pt were loaded for the corresponding samples. The dried complexes were then calcinated at 850 °C for 5 h in air.

$K_2Ti_2O_5$ and metal-doped $K_2Ti_2O_5$ were analyzed by X-ray diffraction (M18XHF, Mac Science Co., Ltd., Yokohama, Japan) in order to assess their purity and crystalline structures. Nickel-filtered Cu K α radiation ($\lambda = 1.5415 \text{ \AA}$) was used with an X-ray gun operated at 40 kV and 200 mA. Diffraction patterns were obtained within the range of $2\theta = 10^\circ - 70^\circ$ with a step size of 0.02° .

2.2. Catalytic Activity Test. Temperature-programmed oxidation (TPO) tests were carried out to evaluate the soot oxidation activities. Reaction tests were performed in a flow-reactor equipment, consisting of a packed bed made of quartz tubes (with an internal diameter of 2 mm). For a typical test, the gas flow rate was adjusted at 100 mL/min. The temperature ramping rate was $10^\circ\text{C}/\text{min}$ at $0-200^\circ\text{C}$, and then $1^\circ\text{C}/\text{min}$ at $200-400^\circ\text{C}$. The mixture of the catalyst (0.1 g) and the soot (0.01 g) was chosen to yield a space velocity of $40\,000 \text{ h}^{-1}$. The contact between catalyst and soot was achieved by grinding in an agate mortar. The reactor was introduced into an electric furnace controlled by a PID temperature controller (Han Kook Electronic Co.). The outlet CO_2 concentration was recorded by a CO_2 analyzer (Chemiluminescence CO_2 analyzer, Model 41C, high level, Thermo Environmental Instruments, Inc.).

3. RESULTS AND DISCUSSION

3.1. Characterization of the Catalysts Doped with Different Metals. Figure 1 shows the XRD profiles of the as-synthesized $Pt/K_2Ti_2O_5$, $Cu/K_2Ti_2O_5$, $Ce/K_2Ti_2O_5$, and $Co/K_2Ti_2O_5$. After comparing with the standard XRD patterns, the supported metals are in the forms of Pt, CuO, CeO_2 , and Co_3O_4 , respectively. The characteristic peaks of Pt are observed at 39.7° , 46.2° , and 67.5° 2θ (JCPDS Powder Diffraction File Card No. 04-0802), CuO at 35.3° and 38.5° (JCPDS Powder Diffraction File Card No. 44-0706), CeO_2 at 28.5° and 33.1° (JCPDS Powder Diffraction File Card No. 43-1002), and Co_3O_4 at 31.3° , 36.8° , and 65.2° (JCPDS Powder Diffraction File Card No. 43-1003), respectively. All the other peaks match well with those of the as-synthesized $K_2Ti_2O_5$. The inset shows the chemical structure of

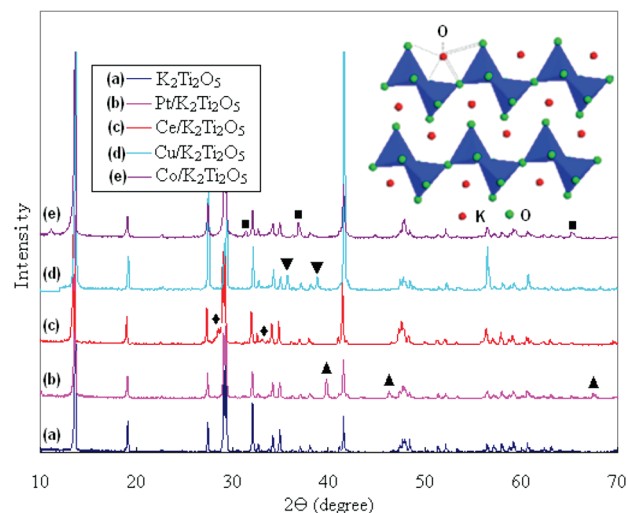


Figure 1. XRD patterns of (a) $K_2Ti_2O_5$, (b) $Pt/K_2Ti_2O_5$, (c) $Ce/K_2Ti_2O_5$, (d) $Cu/K_2Ti_2O_5$, and (e) $Co/K_2Ti_2O_5$. (Peak identification legend: (▲) Pt, (◆) CeO_2 , (▼) CuO, and (■) Co_3O_4 .) The inset shows the atomic structure of $K_2Ti_2O_5$; Ti atoms are located at the center of the tetrahedrons.

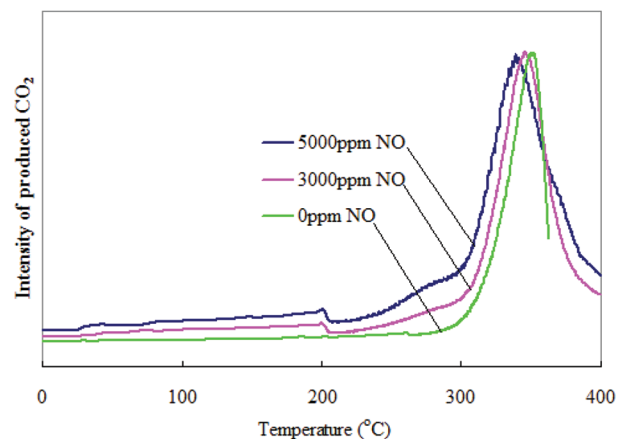


Figure 2. Soot combustion with various NO concentrations over a $K_2Ti_2O_5$ catalyst.

$K_2Ti_2O_5$, which possesses a special configuration of layered structure, in which K atom between the layers has an interesting asymmetrical linkage with eight oxygen anions.³³

3.2. Effects of NO and NO_2 on Soot Oxidation. It is well-known that NO and NO_2 are helpful for soot combustion, especially for Pt-based catalysts.³⁴⁻³⁶ In the presence of Pt, NO can be oxidized to NO_2 , which is a more oxidative reagent than O_2 .³⁷ Therefore, both the effects of NO and NO_2 on soot oxidation were first investigated on $K_2Ti_2O_5$, and the results are shown in Figures 2 and 3. A negligible promotion effect is noticed when NO is added to O_2 , indicating that $K_2Ti_2O_5$, different from Pt catalyst, is not able to oxidize NO to NO_2 . With 5000 ppm of NO, the peak temperature is only decreased by 12°C (see Figure 2). However, NO_2 is observed to be much more effective than O_2 for soot oxidation, as shown in Figure 3. With only 700 ppm NO_2 , two CO_2 desorption peaks, located at 280 and 360°C , were observed during the TPO measurements. By further increasing the concentration of NO_2 to 1200 ppm, the

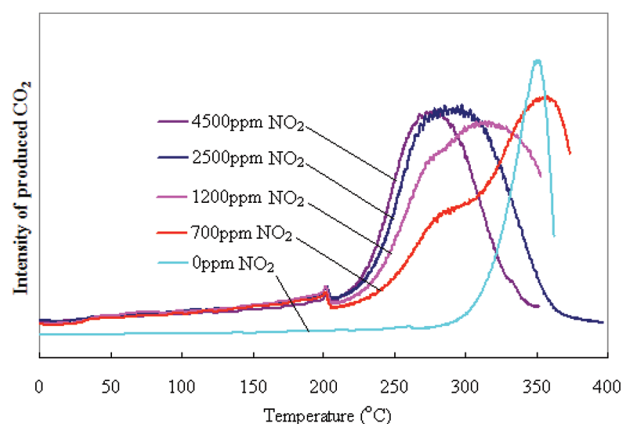


Figure 3. Soot combustion with various NO_2 concentrations over a $\text{K}_2\text{Ti}_2\text{O}_5$ catalyst.

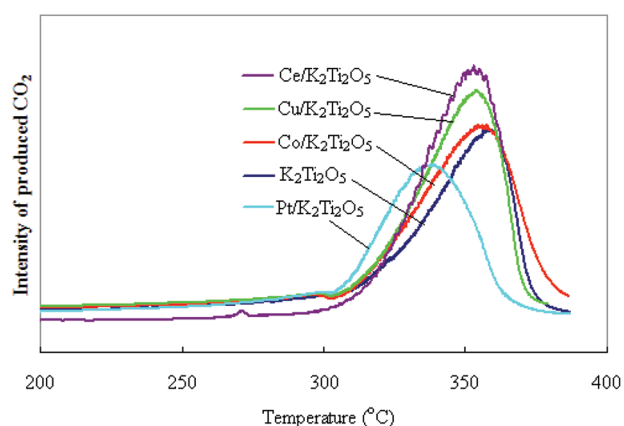


Figure 4. Soot oxidations over $\text{K}_2\text{Ti}_2\text{O}_5$, $\text{Pt}/\text{K}_2\text{Ti}_2\text{O}_5$, $\text{Cu}/\text{K}_2\text{Ti}_2\text{O}_5$, $\text{Co}/\text{K}_2\text{Ti}_2\text{O}_5$, and $\text{Ce}/\text{K}_2\text{Ti}_2\text{O}_5$ catalyst with 5% O_2 .

intensity of the lower-temperature peak is increased, while that of the higher-temperature peak is weakened. Meanwhile, the temperature for the high-temperature peak shifts to a lower value. When the concentration of NO_2 reaches 2500 and 5000 ppm, the high-temperature peak completely disappeared. This result indicates that the low-temperature soot oxidation is attributed to NO_2 , while the high-temperature soot oxidation is attributed to $\text{K}_2\text{Ti}_2\text{O}_5$. It is interesting to note that the soot can be easily oxidized in the presence of NO_2 at ~ 1000 ppm and at temperatures as low as 300°C .

3.3. Effects of the Doping Metals on Soot Oxidation. Figure 4 shows soot oxidation over $\text{K}_2\text{Ti}_2\text{O}_5$ catalysts doped with different metals. In the O_2 system, the doping metals (except platinum) are observed to have negligible promotion effects on soot oxidation. For $\text{Cu}/\text{K}_2\text{Ti}_2\text{O}_5$, $\text{Co}/\text{K}_2\text{Ti}_2\text{O}_5$ and $\text{Ce}/\text{K}_2\text{Ti}_2\text{O}_5$, both the starting temperature (T_s) and the peak temperature (T_p) remain the same as those of the pure $\text{K}_2\text{Ti}_2\text{O}_5$. However, for $\text{Pt}/\text{K}_2\text{Ti}_2\text{O}_5$, the T_p value (335°C) is much lower than that of the pure $\text{K}_2\text{Ti}_2\text{O}_5$ (360°C). The promotion effect of $\text{Pt}/\text{K}_2\text{Ti}_2\text{O}_5$ is mainly due to the high oxidative activity of the supported platinum metal, which has already been considered to be the best catalyst for soot oxidation.^{38,39}

The practical vehicle emissions normally contain a certain amount of NO_x (~ 1000 ppm, over 80% is NO). According to

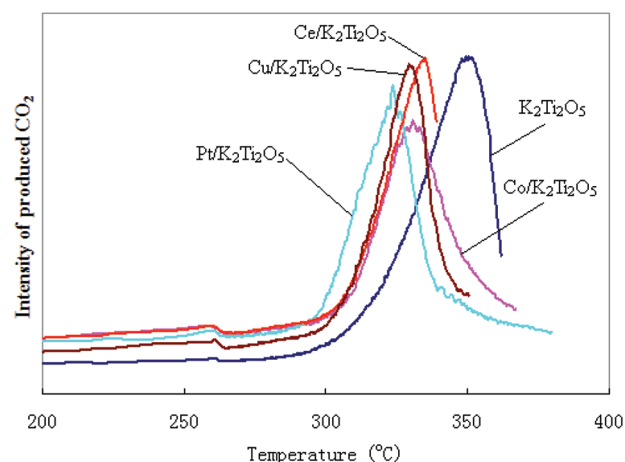


Figure 5. Soot oxidations over $\text{K}_2\text{Ti}_2\text{O}_5$, $\text{Pt}/\text{K}_2\text{Ti}_2\text{O}_5$, $\text{Cu}/\text{K}_2\text{Ti}_2\text{O}_5$, $\text{Co}/\text{K}_2\text{Ti}_2\text{O}_5$, and $\text{Ce}/\text{K}_2\text{Ti}_2\text{O}_5$ with 1000 ppm NO and 5% O_2 .

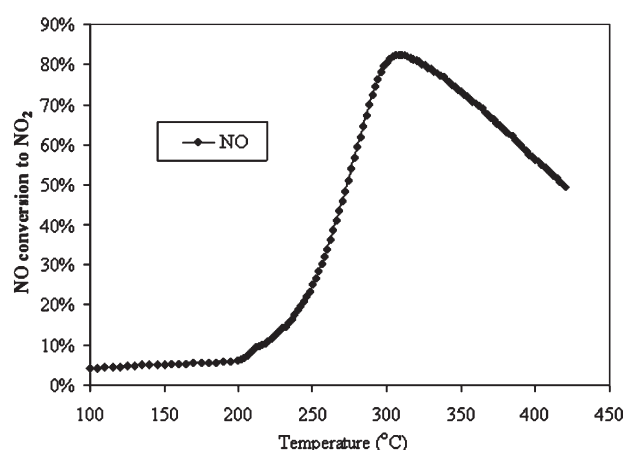


Figure 6. NO oxidation over the $\text{Co}/\text{K}_x\text{Ti}_2\text{O}_5$ catalyst.^{40,41}

recent investigations, the preoxidation of NO to NO_2 will greatly help soot oxidation and decreases the combustion temperature.^{34–36,40} The promotion effect by the doped metals in the NO (1000 ppm) and O_2 system was investigated. The results are shown in Figure 5. Compared to the pure $\text{K}_2\text{Ti}_2\text{O}_5$, all the catalysts doped with metals somehow enhance soot oxidation by decreasing the T_p value, and the activity follows the order of $\text{Pt}/\text{K}_2\text{Ti}_2\text{O}_5$ (324°C) > $\text{Co}/\text{K}_2\text{Ti}_2\text{O}_5$ (331°C) = $\text{Cu}/\text{K}_2\text{Ti}_2\text{O}_5$ (331°C) > $\text{Ce}/\text{K}_2\text{Ti}_2\text{O}_5$ (335°C) > $\text{K}_2\text{Ti}_2\text{O}_5$ (352°C). However, similar to that in the O_2 system, the T_s (ca. 300°C) still remain very high, which is similar to that (300°C) of $\text{K}_2\text{Ti}_2\text{O}_5$. This indicates that the supported metals on $\text{K}_2\text{Ti}_2\text{O}_5$ also have a poor NO oxidation activity in the low-temperature range. Based on this observation, we believe that the soot oxidation activity of $\text{K}_2\text{Ti}_2\text{O}_5$ should be increased by combination with an active NO oxidation catalyst.

3.4. Promotion Effect of the Addition of NO Oxidation Catalyst $\text{Co}/\text{K}_x\text{Ti}_2\text{O}_5$. Recently, we have reported a novel and highly efficient NO oxidation catalyst $\text{Co}/\text{K}_x\text{Ti}_2\text{O}_5$,⁴¹ which can convert over 80% of NO to NO_2 at $\sim 280^\circ\text{C}$ (see Figure 6). The existence of a maximum NO conversion is due to the thermodynamic equilibrium given in eq 1. Please note that, at temperatures of 250 – 300°C , a larger amount of NO_2 can be achieved

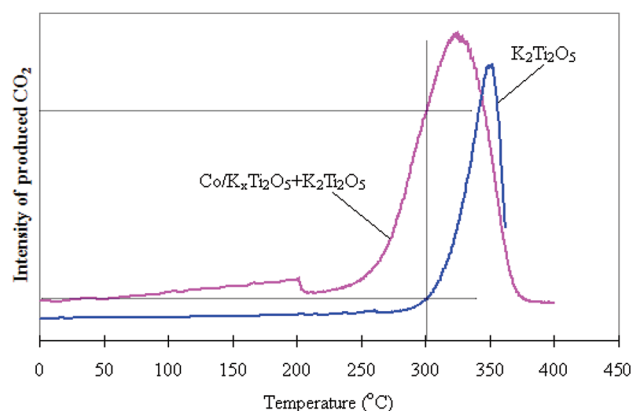


Figure 7. Soot oxidations over $\text{Co}/\text{K}_x\text{Ti}_2\text{O}_5 + \text{K}_2\text{Ti}_2\text{O}_5$ and $\text{K}_2\text{Ti}_2\text{O}_5$ catalyst with 1000 ppm NO and 5% O_2 .

with a conversion rate of 25%–75%. The produced NO_2 can directly oxidize soot to CO_2 at this low-temperature range:

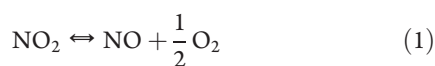


Figure 7 shows the TPO results over the mixture of $\text{Co}/\text{K}_x\text{Ti}_2\text{O}_5$ and $\text{K}_2\text{Ti}_2\text{O}_5$ (1:1 in weight ratio), and $\text{K}_2\text{Ti}_2\text{O}_5$ alone catalysts with NO content of 1000 ppm in the flow gas. By combining a NO oxidation catalyst, both the T_s and T_p values were greatly decreased, from 295 °C to 240 °C and from 352 °C to 326 °C, respectively. It is worthwhile to notice that almost no CO_2 was generated from the pure $\text{K}_2\text{Ti}_2\text{O}_5$ catalyst at 300 °C; however, a significant amount of CO_2 was produced by combining $\text{Co}/\text{K}_x\text{Ti}_2\text{O}_5$ NO oxidation catalyst at the same temperature. It is easy to understand that the promoted soot oxidation activity is due to the produced NO_2 by $\text{Co}/\text{K}_x\text{Ti}_2\text{O}_5$.

4. CONCLUSIONS

Pure $\text{K}_2\text{Ti}_2\text{O}_5$ catalysts and $\text{K}_2\text{Ti}_2\text{O}_5$ catalysts doped with metals ($\text{M}/\text{K}_2\text{Ti}_2\text{O}_5$, where $\text{M} = \text{Pt}, \text{Cu}, \text{Ce},$ and Co) were synthesized and characterized for soot combustion. XRD analysis indicated that the doped metals were in the forms of $\text{Pt}, \text{CuO}, \text{CeO}_2,$ and $\text{Co}_3\text{O}_4,$ respectively. Both the effects of NO and NO_2 on soot combustion were investigated over these catalysts. NO is observed to have limited effects on soot oxidation, even with the catalysts doped with metals. Only $\text{Pt}/\text{K}_2\text{Ti}_2\text{O}_5$ catalysts decreased the T_p value, by 25 °C, with a NO content of 1000 ppm. However, NO_2 is observed to greatly enhance soot oxidation over both $\text{K}_2\text{Ti}_2\text{O}_5$ and $\text{M}/\text{K}_2\text{Ti}_2\text{O}_5$ catalysts. The effect of combining an additional NO oxidation catalyst (1:1 in weight ratio) was also studied, and it showed that the T_p value was greatly decreased, to as low as 326 °C, arising from the NO_2 that was produced by oxidizing NO over a $\text{Co}/\text{K}_x\text{Ti}_2\text{O}_5$ catalyst.

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