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Optimum Preparation Conditions for Alumina Catalyst Containing Cobalt for the Selective Catalytic Reduction of Nitrogen Monoxide with Ethene in Excess Oxygen

Noriyasu OKAZAKI*, Ryuta FUJII, and Akio TADA

Dept. of Applied and Environmental Chemistry, Kitami Institute of Technology, Kitami, Hokkaido 090-8507, JAPAN

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Alumina catalysts containing Co were prepared under various conditions of Co loading, Co salt and calcination temperature to elucidate the factors affecting the activity for the selective reduction of NO with ethene in excess oxygen, and the active Co species. The optimum calcination temperature was 800°C regardless the type of Co salt and the loading of Co, at which formation of CoAl_2O_4 was maximum, formation of Co_3O_4 was minimum and the γ -type structure of alumina was retained. The catalytically active component is CoAl_2O_4 , which acts as a cocatalyst with γ -alumina to complete the reduction of NO.

Keywords

Cobalt aluminate, Nitrogen monoxide, Catalyst preparation, Ethene, Selective catalytic reduction

1. Introduction

Selective catalytic reduction of nitrogen monoxide with hydrocarbons in excess oxygen (SCR-NO) has been investigated extensively as one of the most promising techniques for removing NO in exhaust gases¹⁾. Various catalysts including zeolites, metal oxides, and supported noble metals have been evaluated¹⁾. Alumina shows excellent activity for SCR-NO and its activity is enhanced by addition of transition metal elements^{2)–11)}, among which Co is one of the most effective elements. CoAl_2O_4 formed during calcination is the species involved in the SCR-NO³⁾. Therefore, it is important to know which variables in the preparation procedures affect the formation of CoAl_2O_4 for preparation of highly active catalysts for SCR-NO.

A typical preparation method for Co loaded alumina involves impregnation of alumina with an aqueous solution of cobalt salt followed by drying and calcining at 500–800°C. The loading of Co is usually 0.1–5%. The effects of Co loading, type of Co salt, and calcination temperature on the catalytic properties and type of Co species in the resulting catalyst were examined⁹⁾. Catalyst containing 0.1–0.5% Co prepared by adding cobalt acetate and calcining at 500°C showed the highest activity⁸⁾. Excessive concentrations of surface cobalt ions decreased the activity, probably because of

the promotion of unfavorable side reactions such as the formation of NO. The optimum loading of Co after calcining at different temperatures has not been clarified. The Co species causing the unfavorable side reactions associated with excessive Co has not been identified.

Catalyst calcined at 800°C showed higher activity than catalyst calcined at 500°C^{3),9)}, for catalysts prepared with cobalt acetate and cobalt nitrate with a Co loading of 2%. However, the optimum calcination temperature for catalysts containing different Co loadings, and for catalysts prepared from different cobalt salts has not been investigated. Changes in catalytic activity with calcination at temperatures higher than 800°C are also unknown.

Catalyst prepared from acetate and calcined at 600°C showed higher activity than catalyst prepared from nitrate and calcined at 500°C. The activity of catalyst prepared from chloride has not been assessed.

In the present study, several Co/alumina catalysts were prepared using various Co loadings, Co salts, and calcination temperatures to elucidate how these variables affect the catalytic properties and the formation of Co species.

2. Experimental Method

2.1. Preparation of Catalysts

Alumina (ALOA) was prepared by hydrolyzing aluminum isopropoxide followed by washing with deion-

* To whom correspondence should be addressed.

* E-mail: zaki@catal.chem.kitami-it.ac.jp

Fig. 1 Temperature Dependence of the Catalytic Activity of Co(0.2)/Al₂O₃ Calcined at Various Temperatures

Fig. 2 Temperature Dependence of the Catalytic Activity of Co(5)/Al₂O₃ Calcined at Various Temperatures

ized water, drying at 120°C, and finally calcining at 600°C for 24 h. Co/Al₂O₃ catalysts were prepared by impregnation of the alumina with an aqueous solution of cobalt acetate tetrahydrate, cobalt nitrate hexahydrate, or cobalt chloride hexahydrate with appropriate concentrations. The solvent was removed at 50°C in a rotary evaporator under a water pump vacuum until the solid appeared dry, followed by drying at 120°C for 24 h and calcination at 500°C for 4 h in air. More detailed procedures were reported elsewhere. The catalyst powders were pressed into pellets, followed by crushing, sieving (30–42 mesh), and calcining at 500–1200°C for 4 h in air before use.

Cobalt oxide, Co₃O₄, was prepared by heating cobalt hydroxide at 500°C for 4 h and then at 800°C for 4 h in air. The cobalt hydroxide was prepared from an aqueous solution of cobalt nitrate by hydrolysis with 10% aqueous ammonia to a final pH of 7. The precipitate was filtered, washed, and dried at 120°C for 18 h.

2.2. Characterization of Catalysts

X-Ray powder diffraction patterns were observed with a Rigaku Denki RINT-1200 using nickel filtered CuK α radiation. The specific surface areas of the catalysts was determined by the BET method. UV-VIS reflectance spectra were measured with a Hitachi U-3200 spectrometer equipped with a 60 mm diameter integrating sphere (Hitachi, 130-0632). X-Ray photoelectron spectroscopy (XPS) was performed with a Rigaku Denki XPS-7000 spectrometer, using an AlK α X-ray source operated at 10 kV and 30 mA. The binding energies were corrected using the value of 285.0 eV for the C 1s level resulting from the contaminated carbon. The reproducibilities of the obtained values were within ± 0.2 eV.

2.3. Reaction Procedures

A fixed bed flow reactor was employed for the reduction of NO with ethene at a *W/F* of 0.18 g·s·cm⁻³(catalyst, 0.4 g; total flow rate, 130 cm³·min⁻¹) at

300 to 800°C. The reactant gas (NO, 1000 ppm; O₂, 2.0%; C₂H₄, 500 ppm; He, balance) was passed through the catalyst bed. The outflow gas was analyzed by gas chromatography using two columns packed with 5A molecular sieves (3 m; separation for N₂, O₂, CO, and NO) and Porapak Q (3 m; separation for CO₂, C₂H₄, and N₂O).

3. Results

The present study found that the catalytic activity for the reduction of NO with ethene in excess oxygen varied with the Co loading, calcination temperature, and the type of cobalt salt used to prepare the catalyst.

3.1. Effects of Co Loading and Calcination Temperature on Co/Al₂O₃ Prepared from Cobalt Acetate

Figure 1 shows the effect of the calcination temperature on the activity of Co/alumina, measured as conversion of NO to N₂, prepared from cobalt acetate and containing 0.2 wt% Co. The activities are plotted against the reaction temperature for the catalysts calcined at different temperatures. As a reference, the activities of alumina calcined at 800°C and without addition of Co are also shown. The activity of Co/alumina containing 0.2 wt% Co was much higher than that of alumina without Co loading, and remained unchanged for calcination temperatures in the range 500–800°C, but the activity decreased after calcination at 1000°C.

Figure 2 shows the effect of the calcination temperature on the activity of Co/alumina containing 5 wt% Co. The activity of Co/alumina containing 5 wt% Co was low after calcination at 500°C, but the activity became higher at calcination temperatures in the range 700–800°C, than decreased after calcination at 1000°C and was almost zero after calcination at 1200°C.

The effect of the calcination temperature on the

Fig. 3 Calcination Temperature Dependence of the Catalytic Activity of Co/Al₂O₃

activity of Co/alumina containing 1 wt% Co was intermediate between those on Co/alumina containing 0.2 wt% Co and Co/alumina containing 5 wt% Co. High activity for Co/alumina containing 20 wt% Co was obtained only after calcination at 800°C, whereas activity was negligible after calcination at 500°C.

Figure 3 shows the catalytic activity at a reaction temperature of 500°C as a function of the calcination temperature for Co/alumina catalyst containing different Co loadings. The activity depended strongly on the Co loading for calcination temperatures in the range 500–800°C. The increase in activity with an increase in calcination temperature was greater for a larger Co loading. The maximum activities were obtained after calcination at 800°C for all catalysts. The activity decreased for all catalysts with calcination temperature above 800°C.

3.2. Effects of Co Loading and Calcination Temperature on Co/Al₂O₃ Prepared from Cobalt Nitrate and Cobalt Chloride

The effect of calcination temperature on the catalytic activity varied with the Co loading. Figure 4 shows the catalytic activity as a function of the reaction temperature for catalysts containing 0.2 wt% Co prepared from different cobalt salts and calcined at 500°C (Fig. 4 a) and 800°C (Fig. 4 b)). Neither the calcination temperature nor the type of cobalt salt affected the activity of the catalysts containing 0.2 wt% Co.

Figure 5 shows the catalytic activity as a function of the reaction temperature for catalysts containing 5 wt% Co prepared from cobalt nitrate and cobalt chloride and calcined at 500°C and 800°C. The catalysts prepared from cobalt nitrate and cobalt chloride by calcination at 500°C exhibited much lower activities than the catalyst containing 5 wt% Co prepared from cobalt acetate. However, the activities of these catalysts were remarkably increased by calcination at 800°C and the catalytic activities were essentially the same regardless of the

Fig. 4 Temperature Dependence of the Catalytic Activity of Co(0.2)/Al₂O₃ Prepared from Various Co Salts and Calcined at a) 500°C and b) 800°C

Fig. 5 Temperature Dependence of the Catalytic Activity of Co(5)/Al₂O₃ Prepared from Various Co Salts

type of cobalt salt.

3.3. Effects of Co Loading and Calcination Temperature on the Conversion of Ethene in the Selective Reduction of NO

Oxidation of ethene to CO_x also occurs in the reduction of NO with ethene in excess oxygen was also affected by the Co loading and the calcination temperature. Oxidation of ethene at lower temperature indicates lower catalytic activity for SCR-NO. Figures 6 a) and b) show the conversions of ethene to CO_x with reaction temperature for the catalysts prepared from cobalt acetate and calcined at 500°C and 800°C, respectively. Conversion of ethene to CO_x occurred at lower temperatures for catalysts with higher Co loading calcined at 500°C. However, the conversion of ethene to CO_x occurred at similar temperatures regardless of the Co loading for the catalysts calcined at 800°C, and close to the temperature for the catalyst containing 0.2 wt% Co calcined at 500°C.

Fig. 6 Temperature Dependence of the Catalytic Activity of Co/Al₂O₃ Calcined at a) 500°C and b) 800°C

Fig. 7 Diffuse Reflectance Spectra of Co(A, 5)/Al₂O₃ Calcined at Various Temperatures

3. 4. Active Co Species on Alumina

Figure 7 shows the UV-VIS spectra of the catalysts containing 5 wt% Co prepared from cobalt acetate and calcined at different temperatures together with the spectra of Co_3O_4 and $CoAl_2O_4$. Co_3O_4 has weak broad peaks at 380 nm, and 650 nm. $CoAl_2O_4$ has triple peaks in the range 500-650 nm, and a broad shoulder in the range 300-400 nm.

The catalyst calcined at 300°C had a spectrum similar to that of Co_3O_4 , and showed no peaks characteristic of $CoAl_2O_4$. The peaks ascribed to $CoAl_2O_4$ began to appear at the calcination temperature of 500°C, and became stronger at higher calcination temperatures. The triple peaks became maximum at the calcination temperature of 800°C. The shoulder peak in the range 300-400 nm developed at calcination temperatures of 1200°C and 1400°C. Clearly, Co_3O_4 was converted into $CoAl_2O_4$ by calcination, beginning at below 500°C and increasing until about 800°C at which temperature most Co_3O_4 was converted to $CoAl_2O_4$.

Figure 8 shows XPS spectra of the Co 2p core level for the catalysts calcined at different temperatures. The deconvoluted curves resulting from computer peak fitting are also shown with the binding energies for Co_3O_4 and $CoAl_2O_4$. The peak intensity corresponding to Co_3O_4 was decreased by raising the calcination

temperature from 500 to 800°C, and the peak for Co_3O_4 was not detected after calcination at 1000°C. The XPS findings also show that Co_3O_4 was converted to $CoAl_2O_4$ by calcination, and converted almost completely after calcination at 1000°C for the catalyst containing 5 wt% Co prepared from cobalt acetate.

The fractions of Co_3O_4 and $CoAl_2O_4$ after calcination at a certain temperature varied with the Co loading. Figure 8 shows the change in the fraction of $CoAl_2O_4$ of total Co species in Co/alumina calcined at 500°C and 800°C as a function of the Co loading. The fraction of $CoAl_2O_4$ markedly decreased with increased Co loading for Co/alumina calcined at 500°C, to 0.92 for the catalyst containing 0.2 wt% Co, and 0.43 for the catalyst containing 20 wt% Co. In contrast, the fraction of $CoAl_2O_4$ did not decrease much with the Co loading for the catalysts calcined at 800°C, exceeding 0.94 regardless of the Co loading. Figure 9 shows the changes in fraction of $CoAl_2O_4$ in the catalysts prepared from cobalt acetate and cobalt nitrate containing 5 wt% Co as a function of the calcination temperature. The fraction of $CoAl_2O_4$ in the catalyst prepared from cobalt nitrate was only 0.35, whereas that in the catalyst prepared from cobalt acetate exceeded 0.85 after calcination at 500°C. In contrast, the fraction of $CoAl_2O_4$ was the same for both catalysts after calcination at 800°C. Conversion of Co_3O_4 into $CoAl_2O_4$ is

Fig. 8 Co $2p_{3/2}$ XPS Spectra of Co(A, 5)/Al $_2$ O $_3$ Calcined at Various Temperatures

greater for catalyst prepared from cobalt acetate than for catalyst prepared from cobalt nitrate. The conversion of Co $_3$ O $_4$ or CoCl $_2$ into CoAl $_2$ O $_4$ could not be measured for the catalyst prepared from cobalt chloride because the peak of CoCl $_2$ was superimposed on the peak of CoAl $_2$ O $_4$.

Table 1 lists the crystal phases of the various catalysts together with the surface areas. The surface area gradually decreased with higher calcination temperature regardless of the Co loading and Co source. A substantial decrease was observed after calcination at 1000°C and 1200°C. The surface area also decreased for the catalyst containing 20 wt% Co.

The crystalline structure of alumina was the γ -type after calcination up to 800°C. α -Alumina was present with γ -alumina after calcination at 1000°C, and only α -alumina was present after calcination at 1200°C.

Only Co $_3$ O $_4$ was found after calcination up to 500°C for the catalysts prepared from cobalt acetate and cobalt nitrate, and only CoCl $_2$ after calcination at 500°C for the catalyst prepared from cobalt chloride. However, only CoAl $_2$ O $_4$ was present regardless of the source of

Fig. 9 Fraction of CoAl $_2$ O $_4$ in Co Species (CoAl $_2$ O $_4$ and Co $_3$ O $_4$) of Co(A)/Al $_2$ O $_3$ as a Function of Cobalt Loading

Fig. 10 Fraction of CoAl $_2$ O $_4$ in Co Species (CoAl $_2$ O $_4$ and Co $_3$ O $_4$) of Co/Al $_2$ O $_3$ as a Function of Calcination Temperature

cobalt after calcination at 800, 1000 and 1200°C.

4. Discussion

The results of the present study suggest that CoAl $_2$ O $_4$ is the Co-species involved in the catalytic activity for the reduction of NO with ethene^{3),11)}. The formation of CoAl $_2$ O $_4$ on alumina is affected by the loading of Co, the calcination temperature, and the source of Co. The Co/alumina catalyst can be prepared to maximize the fraction of CoAl $_2$ O $_4$ among the total Co species.

Reduction of NO with hydrocarbons in excess oxygen proceeds by the successive steps of (1) NO + 1/2 O $_2$ → NO $_2$ and (2) NO $_2$ + HC → N $_2$ + CO $_x$ + H $_2$ O. The reaction (3) HC + O $_2$ → CO $_x$ + H $_2$ O also occurs, but is not desirable because ethene is unproductively consumed. CoAl $_2$ O $_4$ actively catalyzes reaction (1), but not reaction (3)¹³⁾, whereas Co $_3$ O $_4$ actively catalyzes

Table 1 Surface Area and X-Ray Diffraction Patterns of Co/Al₂O₃ Catalysts

Co loading [%]	Co source	Calc. temp [°C]	Surface area [m ² ·g ⁻¹]	X-ray diffraction patterns
5	acetate	300	195	γ -Al ₂ O ₃ , Co ₃ O ₄
5	acetate	500	180	γ -Al ₂ O ₃ , Co ₃ O ₄
5	acetate	600	166	γ -Al ₂ O ₃ , Co ₃ O ₄ , CoAl ₂ O ₄
5	acetate	700	156	γ -Al ₂ O ₃ , CoAl ₂ O ₄
5	acetate	800	138	γ -Al ₂ O ₃ , CoAl ₂ O ₄
5	acetate	1000	69	γ -Al ₂ O ₃ , α -Al ₂ O ₃ , CoAl ₂ O ₄
5	acetate	1200	4	α -Al ₂ O ₃ , CoAl ₂ O ₄
5	nitrate	500	209	γ -Al ₂ O ₃ , Co ₃ O ₄
5	nitrate	800	120	γ -Al ₂ O ₃ , CoAl ₂ O ₄
5	chloride	500	164	γ -Al ₂ O ₃ , CoCl ₂
5	chloride	800	129	γ -Al ₂ O ₃ , CoAl ₂ O ₄
0.2	acetate	500	195	—
1	acetate	500	192	—
2.5	acetate	500	189	—
0.2	acetate	800	144	—
1	acetate	800	143	—
2.5	acetate	800	141	—
20	acetate	800	109	γ -Al ₂ O ₃ , CoAl ₂ O ₄

reaction (3)^{12,13}. Therefore, the CoAl₂O₄ species are essential to the reduction of NO with ethene in excess oxygen as controlled by reaction (1).

The fraction of CoAl₂O₄ was high in catalysts with low Co loading even after calcination at low temperature. Co/alumina catalyst containing 0.2 wt% Co included 0.92 of Co species as CoAl₂O₄ after calcination at 500°C, and 0.98 as CoAl₂O₄ after calcination at 800°C. Therefore, the optimum calcination temperature range is wide for Co/alumina containing 0.2 wt% Co.

In contrast, the fraction of CoAl₂O₄ was low in catalysts with high Co loading after calcination at low temperature, and a large fraction of Co species remained as Co₃O₄. Co/alumina catalyst containing 20 wt% Co included only 0.42 of Co species as CoAl₂O₄ after calcination at 500°C, which increased to 0.95 after calcination at 800°C. Therefore, the optimum calcination temperature range was narrow for the Co/alumina containing 20 wt% Co. Co/alumina catalysts containing between 0.2 wt% Co and 20 wt% Co showed intermediate relationships between the catalytic activity and the calcination temperature.

Conversion of Co₃O₄ into CoAl₂O₄ is affected not only by the Co loading but also by the type of Co salt used as the Co source. Calcination temperature and the type of Co salt had little effect for the Co/alumina catalysts containing 0.2 wt% Co, but much greater effects for the catalysts containing a larger amount of Co. Catalysts containing 5 wt% Co and calcined at 500°C showed catalytic activities in the order: Co/alumina prepared from cobalt acetate > Co/alumina prepared from CoCl₂ > Co/alumina prepared from Co(NO₃)₂. The fraction of CoAl₂O₄ was larger for the catalyst prepared from Co acetate than for the catalyst

prepared from Co(NO₃)₂. The Co species in the catalyst prepared from CoCl₂ were mostly in the form of CoCl₂. By raising the calcination temperature, the catalytic activities of the Co/alumina catalysts prepared from different Co salts became the same, indicating that most Co species were converted into CoAl₂O₄ by calcination at 800°C regardless of the Co salt.

The relationships between the Co loading, calcination temperature, and catalytic activity were evaluated by measuring the ethene conversion. Catalysts with high Co loading and low calcination temperature converted ethene by SCR-NO at low temperatures. In contrast, catalysts with high Co loading and low calcination temperature, converted ethene by reaction with oxygen and not with NO due to the considerable remaining Co₃O₄. By raising the calcination temperature to 800°C, Co₃O₄ was converted into CoAl₂O₄ and the catalyst showed a high activity for SCR-NO.

The fraction of CoAl₂O₄ increases with calcination temperatures exceeding 800°C as shown by UV spectroscopy and XPS. However, the catalytic activity decreases as the calcination temperature exceeds 800°C, because α -alumina begins to appear. Only the α -alumina phase is detected by XRD after calcination at 1200°C, and the catalytic activity becomes negligibly small. Therefore, γ -alumina is required for catalytic activity for the reduction of NO. CoAl₂O₄ promotes the oxidation of NO to NO₂ (reaction (1)), and γ -alumina promotes the reaction of NO₂ with ethene to N₂ (reaction (2)). Regardless of the Co loading and the type of Co salt, the highest catalytic activity was obtained by calcination at 800°C, at which the fraction of CoAl₂O₄ was sufficiently high, the fraction of Co₃O₄ remained negligibly small, and the γ -type alumina was retained. However, the possibility that the low activi-

ty of the catalyst calcined at 1200°C was simply caused by a decrease in surface area cannot be excluded.

5. Conclusions

The formation of CoAl_2O_4 as the active species in the catalyst is affected by the Co loading, type of Co salt, and calcination temperature.

(1) The optimum Co loading varies with the calcination temperature. At a low calcination temperature of 500°C, 0.2 wt% Co provided the highest activity. At a calcination temperature of 800°C, all catalysts containing Co in the range 0.2-20 wt% had high activities.

(2) The optimum calcination temperature varies with the Co loading. The optimum calcination temperature range for low loading is wide; catalyst loaded with 0.2 wt% Co had high activities in the calcination temperature range 500-800°C whereas catalyst loaded with 20 wt% Co had high activity only after calcination at 800°C.

(3) The optimum calcination temperature range varies with the type of cobalt salt. The activity depended strongly on the calcination temperature for the catalysts prepared from cobalt nitrate and cobalt chloride but weakly for those prepared from cobalt acetate. All catalysts prepared from different cobalt salts showed the highest activity after calcination at 800°C.

(4) γ -Alumina is probably essential for an active catalyst; as γ -alumina promotes the reaction of NO_2 with ethene to form N_2 .

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要 旨

酸素過剰雰囲気下におけるエチレンによる NO 選択還元反応 —コバルト添加アルミナ触媒の最適調製条件—

岡崎 文保, 藤井 竜太, 多田 旭男

北見工業大学化学システム工学科, 090-8507 北海道北見市公園町 165

酸素過剰雰囲気下でエチレンを還元剤に用いた一酸化窒素の選択還元反応において、触媒活性に影響を及ぼす要因、および活性に関わるコバルト種を明らかにするために、コバルト添加量、コバルト出発塩の種類、および触媒焼成温度など、調製条件の異なるコバルト添加アルミナの触媒活性を詳細に検討した。その結果、コバルト出発塩の種類とその添加量によらず最

適焼成温度は 800°C であること、この焼成温度ではアルミナが γ 型の構造を保持したまま、アルミナ上でコバルトアルミネートの生成が容易に進行し、酸化コバルトの量が最小となることがわかった。これは γ -アルミナの NO 還元活性を向上させる活性なコバルト種はコバルトアルミネートであることを示唆する。