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Article

Alumina-carbon nanotube supports for sulfur-resistant regenerable Pt-based catalysts in NO_x reduction

Lilong Jiang *, Yuling Wang, Xian Liu, Yanning Cao, Kemei Wei

National Engineering Research Center of Chemical Fertilizer Catalyst, Fuzhou University, Fuzhou 350002, Fujian, China

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ABSTRACT

Alumina-carbon nanotube (Al₂O₃-CNT) supports were prepared by the decomposition of acetylene on Al₂O₃. The subsequent impregnation of Pt and Ba yielded Pt/Ba/Al₂O₃-CNT. Pt/Ba/Al₂O₃-CNT was characterized by X-ray diffraction, scanning and transmission electron microscopies, N₂ physisorption, X-ray photoelectron spectroscopy, and in-situ diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS). The relationship between the structure and properties of Pt/Ba/Al₂O₃-CNT was investigated. Pt/Ba/Al₂O₃-CNT exhibited much higher resistance to SO₂ poisoning and better regeneration ability than Pt/Ba/Al₂O₃, during NO_x reduction in the presence of SO₂. In-situ DRIFTS spectra suggested that SO₂ had no effect on the NO_x storage and reduction process.

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1. Introduction

Nitrogen oxides (NO_x) are emitted from stationary sources and automobiles, and pose a threat to environmental and human health [1]. Increasing car numbers and NO_x emission regulations have encouraged the development of technology to eliminate NO_x from lean-burn exhaust. Traditional three-way catalysts are inefficient for NO_x abatement under lean-burn conditions [2,3]. NO_x storage-reduction (NSR) is a promising technology for NO_x removal in lean-burn engines. NSR catalysts usually contain precious metals, alkali or alkaline earth metals, and high surface area supports [3]. Pt/Ba/Al₂O₃ is a model NSR catalyst, and has been the subject of considerable experimental and theoretical investigation [4–7]. One of its disadvantages is deactivation due to sulfur poisoning [8]. Sulfur present in fuel and lubricants can poison Pt/Ba/Al₂O₃ by forming stable barium sulfate, which blocks storage sites and platinum surfaces. The recovery of the catalyst by sulfur removal requires high

temperature, and successive regeneration usually causes sintering of precious metals and storage materials. The solid state reaction between Ba and Al₂O₃ may also result in the formation of inactive BaAlO₂ [9].

Improving the sulfur resistance and thermal stability of NSR catalysts has received much attention. Supports significantly affect the catalytic activity, and using composite supports can improve NSR performance. For example, blending TiO₂ and Al₂O₃ can suppress sulfur deposition, and enhance the NO_x storage capacity of sulfur-aged catalysts [10–12]. An acceptable balance between thermal resistance and sulfur tolerance can be achieved in TiO₂-ZrO₂ supports, leading to an increased NO_x storage ability of sulfur-aged NSR catalysts [13].

Carbon nanotubes (CNTs) have been extensively used as supports in catalytic reactions, because of their favorable structure and unique properties [14,15]. The large surface area and confinement effect in CNTs may prevent the agglomeration of metal components, resulting in high catalytic activity [16,17].

* Corresponding author. Tel: +86-591-83731234-8201; Fax: +86-591-83707796; E-mail: jll@fzu.edu.cn

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Luo et al. [18] attributed the higher NO conversion over Rh/CNTs than Rh/ γ -Al₂O₃ to the superior stabilization of Rh⁰ on the CNTs. Iron-copper oxides supported on TiO₂ and CNTs reportedly exhibit high resistance against sulfur poisoning, and excellent NO_x elimination performance at low temperature [19]. In our continuous effort to develop sulfur-resistant NSR catalysts [20], herein we introduce CNTs into Pt/Ba/Al₂O₃ to form a Pt/Ba/Al₂O₃-CNTs catalyst. Its structure, NSR performance, and the effects of SO₂ concentration and regeneration time on NSR are investigated. The role of CNTs in the Pt/Ba/Al₂O₃-CNTs catalyst is also investigated.

2. Experimental

2.1. Preparation of Pt/Ba/Al₂O₃-CNTs and Pt/Ba/Al₂O₃

Alumina was synthesized according to the literature methods [21]. CNTs were prepared by chemical vapor deposition (CVD). An aqueous solution of molybdenum nitrate was impregnated on Al₂O₃, subsequently placed in a porcelain boat in a tube reactor, pretreated in H₂ at 500 °C for 2 h, and heated to 750 °C in an Ar flow. A gas mixture of C₂H₂ (20 mL/min) and H₂ (100 mL/min) was subsequently introduced for 0.5 h, followed by an H₂ flow for 2 h to remove excess carbon, before cooling to room temperature in Ar. The product was purified by soaking in 30% H₂O₂ for 48 h, dried at 100 °C overnight, and calcined in air at 550 °C for 4 h to yield Al₂O₃-CNTs.

Ba (16%) and Pt (1%) were dispersed on Al₂O₃-CNTs by conventional impregnation, using aqueous Ba(OAc)₂ and H₂PtCl₆·6H₂O solutions, respectively. The impregnated sample was then dried at 120 °C overnight, and calcined in air at 500 °C for 4 h. The obtained catalyst was denoted as Pt/Ba/Al₂O₃-CNTs. For comparison, Pt/Ba/Al₂O₃ with the same amount of Ba and Pt was also prepared by the same procedure as Pt/Ba/Al₂O₃-CNTs, except that Al₂O₃ was used as the support.

2.2. Characterization

Powder X-ray diffraction (XRD) was performed on a Rigaku-DMax 2500PC diffractometer, using Cu-K α radiation (λ = 0.15406 nm). XRD patterns were recorded at 2θ = 10°–80°. N₂ physisorption measurements were carried out using a Micromeritics ASAP 2020 apparatus. Samples were degassed in vacuo at 180 °C for at least 6 h before measurements. Pore size distributions were obtained from the isotherm adsorption branch, using the Barrett Joyner Halenda (BJH) model. X-ray photoelectron spectroscopy (XPS) was performed on a Physical Electronics Quantum 2000 instrument, equipped with a monochromatic Al-K α source ($K\alpha$ = 1486.6 eV) and a charge neutralizer. Transmission electron microscopy (TEM) was performed on a FEI Tecnai G² F20 microscope, operated at 200 kV. Scanning electron microscopy (SEM) was performed on a Hitachi S-4800 microscope, operated at 5.0 kV. Inductively coupled plasma (ICP) spectroscopy was performed on a JY Ultima2 spectrometer.

In-situ diffuse reflectance infrared Fourier transform spec-

tra (DRIFTS) were recorded on a Nicolet Nexus FT-IR spectrometer over the 4000–650 cm⁻¹ range, from 32 scans at 4 cm⁻¹ resolution. Approximately 50 mg of sample in a DRIFTS cell was pretreated with He at the testing temperature for 30 min, and the recorded spectrum was taken as the background. A gas mixture of NO (500 × 10⁻⁶) and 2% O₂ was introduced into the sample cell, and spectra were collected. An additional SO₂ (100 × 10⁻⁶) was then introduced into the sample cell, and spectra were collected to follow the evolution of different surface species. The regeneration of Pt/Ba/Al₂O₃-CNTs was performed with 3.5% H₂-96.5% Ar, after samples were exposed to SO₂ (100 × 10⁻⁶) at 400 °C for 1 h. DRIFTS spectra were recorded after different time. The final differential sample spectra were calculated using the Kubelka-Munk function.

2.3. Catalytic activity measurements

Catalytic tests were carried out in a stainless steel fixed bed flow reactor (8 mm internal diameter). Prior to each test, the sample (0.5 g) was pretreated in a N₂ flow (30 mL/min) at 500 °C for 1 h to remove any adsorbed species, and then cooled to the test temperature. The feed gas was introduced using mass-flow controllers, at a total flow rate of 600 mL/min. The NO_x concentration was measured using an on-line chemiluminescence NO-NO₂-NO_x analyzer (model 42i-HL, Thermo Scientific). After measurement, the catalysts were sulfur-aged by exposing to a feed gas containing SO₂ (0–250 × 10⁻⁶) for 45 min. Sulfur-aged Pt/Ba/Al₂O₃-CNTs and Pt/Ba/Al₂O₃ were denoted as Pt/Ba/Al₂O₃-CNTs-SO₂ and Pt/Ba/Al₂O₃-SO₂, respectively. The regeneration ability of the sulfur-aged samples was tested in 3.5% H₂ and 96.5% Ar at 400 °C.

3. Results and discussion

3.1. SEM and TEM results

The morphology of Al₂O₃-CNTs is shown in Fig. 1. The SEM image revealed that the CNTs were coiled Fig. 1(a). A typical nanotube was analyzed by TEM Fig. 1(b), and its outer and inner diameters were *ca.* 48 and 43 nm, respectively. A few alumina particles were also observed in the SEM image.

3.2. Textural properties

The textural properties of the supports and corresponding catalysts are summarized in Table 1. The specific surface area of Al₂O₃-CNTs was 416 m²/g, which was much larger than that

Fig. 1. SEM (a) and TEM (b) images of Al₂O₃-CNTs.

Table 1

Textural properties of the samples.

Sample	BET surface area (m ² /g)	Pore volume (cm ³ /g)	Average pore radius (nm)
Al ₂ O ₃	266	0.56	4.87
Al ₂ O ₃ -CNTs	416	0.66	6.30
Pt/Ba/Al ₂ O ₃	216	0.41	6.26
Pt/Ba/Al ₂ O ₃ -CNTs	389	0.62	3.54

of Pt/Ba/Al₂O₃. The specific surface area and pore volume of Pt/Ba/Al₂O₃-CNTs and Pt/Ba/Al₂O₃ decreased after impregnation by Pt and Ba, while their average pore radii increased. ICP analyses indicated that their Pt and Ba contents were 0.86% and 10.84%, respectively, which were lower than their loading amounts. The content of molybdenum (0.00035%) was negligible, suggesting that molybdenum was completely removed during CNT preparation.

3.3. NSR performance

The NSR performance of Pt/Ba/Al₂O₃-CNTs was initially evaluated using the relative NO_x storage capacity and NO_x conversion, in the absence of SO₂. As shown in Fig. 2, the relative NO_x storage capacity and NO_x conversion over Pt/Ba/Al₂O₃-CNTs were similar to those over Pt/Ba/Al₂O₃. Temperature had no obvious effect on the relative NO_x storage capacity (2 min), which was >90% over the range of tested tempera-

tures. However, NO_x conversion was enhanced as the temperature increased from 200 to 350 °C. For example, NO_x conversions at 200 °C and 350 °C were 22.89% and 91.29%, respectively.

The effect of sulfur poisoning on the NO_x reduction over Pt/Ba/Al₂O₃-CNTs and Pt/Ba/Al₂O₃ was investigated in the presence of SO₂ (0–250 × 10⁻⁶), as shown in Fig. 3. When fresh Pt/Ba/Al₂O₃-CNTs and Pt/Ba/Al₂O₃ were reduced by 3.5% H₂ at 300 °C for 1 min, the outlet NO_x concentrations were 0.89 × 10⁻⁶ and 2.54 × 10⁻⁶, respectively. When SO₂ (50 × 10⁻⁶) was introduced into feed gas, the outlet NO_x concentration increased to 5.26 × 10⁻⁶ and 107 × 10⁻⁶, respectively, suggesting that Pt/Ba/Al₂O₃-CNTs and Pt/Ba/Al₂O₃ were deactivated by SO₂. The deactivation rate was related to the SO₂ concentration. When the SO₂ concentration was increased to 250 × 10⁻⁶, a significant decrease in NO_x reduction rate was observed, and the outlet NO_x concentrations for Pt/Ba/Al₂O₃-CNTs and Pt/Ba/Al₂O₃ reached 207 × 10⁻⁶ and 375 × 10⁻⁶, respectively. SO₂ had a more pronounced effect on Pt/Ba/Al₂O₃ than Pt/Ba/Al₂O₃-CNTs. Deactivation from SO₂ was ascribed to the strong affinity of barium with SO₃. This formed stable barium sulfate [22], and restricted the contact between Pt and NO₂. Literature reports [16,17] indicate that CNTs exhibit good NO_x adsorption and oxidation resistance. The oxidation of SO₂ to SO₃ may have been inhibited in the SO₂ atmosphere, which decreased the formation of BaSO₄ during NO_x reduction.

NSR catalyst regeneration is related to time, so the effect of

Fig. 2. Relative NO_x storage capacity (a) and NO_x conversion (b) over Pt/Ba/Al₂O₃-CNTs and Pt/Ba/Al₂O₃ catalysts.

Fig. 3. Outlet NO_x concentration at 300 °C for the exposure of Pt/Ba/Al₂O₃-CNTs (a) and Pt/Ba/Al₂O₃ catalysts (b) to different SO₂ concentrations.

Fig. 4. Outlet NO_x concentration at 300 °C for fresh, sulfur-aged SO₂ (150×10^{-6}) and regenerated Pt/Ba/Al₂O₃-CNTs (a) and Pt/Ba/Al₂O₃ catalysts (b).

regeneration time on the sulfur-aged catalysts was investigated. As shown in Fig. 4, the NO_x reduction ability could be somewhat recovered after the sulfur-aged samples were regenerated. For example, the outlet NO_x concentrations were 22.3×10^{-6} and 118×10^{-6} , respectively, after Pt/Ba/Al₂O₃-CNTs and Pt/Ba/Al₂O₃ were sulfur-aged by SO₂ (150×10^{-6}) and then reduced by 3.5% H₂ for 1 h. Pt/Ba/Al₂O₃-CNTs exhibited a higher resistance to SO₂ poisoning and better regeneration ability than Pt/Ba/Al₂O₃.

3.4. XRD results

To further investigate the effect of sulfur poisoning on Pt/Ba/Al₂O₃-CNTs and Pt/Ba/Al₂O₃, fresh and sulfur-aged samples were investigated by XRD and XPS. XRD patterns indicated the superior sulfur tolerance of Pt/Ba/Al₂O₃-CNTs. Figure 5 shows that after fresh samples were treated by SO₂ (150×10^{-6}) for (0.5 h), strong peaks at $2\theta = 26.0^\circ$, 28.7° , and 42.6° (characteristic of BaSO₄) were observed in the XRD pattern of Pt/Ba/Al₂O₃-SO₂ [12]. Only weak BaSO₄ peaks were observed in that of Pt/Ba/Al₂O₃-CNTs-SO₂. No Pt species peaks were

observed, suggesting that Pt species were either finely dispersed or too small to be detected by XRD [23].

3.5. XPS results

To evaluate the sulfur content accumulated on the catalyst surface, SO₂-treated and H₂-regenerated catalysts were investigated by XPS. The XPS peak at *ca.* 169.4 eV was assigned to sulfate, arising from the uptake of sulfur during SO₂ exposure (Fig. 6) [4]. The sulfur content of Pt/Ba/Al₂O₃-CNTs-SO₂ was lower than that of Pt/Ba/Al₂O₃-SO₂ (Table 2). Sulfur peaks were clearly observed in the XPS spectrum of Pt/Ba/Al₂O₃-SO₂-H₂, but had largely disappeared in the spectrum of Pt/Ba/Al₂O₃-CNTs-SO₂-H₂. These results indicated the higher sulfur tolerance and regeneration ability of Pt/Ba/Al₂O₃-CNTs, compared with Pt/Ba/Al₂O₃.

3.6. In-situ DRIFTS results

To further investigate the effect of SO₂ on NSR in Pt/Ba/Al₂O₃-CNTs, in-situ DRIFTS experiments were carried out. Fig.

Fig. 5. XRD patterns for Pt/Ba/Al₂O₃-CNTs (1), Pt/Ba/Al₂O₃-CNTs-SO₂ (2), Pt/Ba/Al₂O₃ (3), and Pt/Ba/Al₂O₃-SO₂ (4).

Fig. 6. XPS S 2p spectra of Pt/Ba/Al₂O₃-CNTs and Pt/Ba/Al₂O₃, after being sulfur-aged SO₂ (150×10^{-6}) and regenerated.

Table 2

Surface sulfur content determined by XPS.

Sample	S 2p (%)
Pt/Ba/Al ₂ O ₃ -CNTs-SO ₂	0.61
Pt/Ba/Al ₂ O ₃ -SO ₂	2.08
Pt/Ba/Al ₂ O ₃ -CNTs-SO ₂ -H ₂	0.14
Pt/Ba/Al ₂ O ₃ -SO ₂ -H ₂	1.04

ure 7(a) shows that when Pt/Ba/Al₂O₃-CNTs was exposed to NO (500×10^{-6})-2% O₂ in Ar at 200 °C for 0.5h, the characteristic band of bidentate nitrite at ca. 1247 cm⁻¹ was observed [24]. The band intensity gradually weakened with increasing temperature, and the nitrate peak at 1334 cm⁻¹ became progressively more intense. A broad peak at 1635 cm⁻¹ was assigned to bridging/chelating bidentate nitrate at surface Ba sites [25], and a blue shift was observed with increasing temperature [26]. Thus, the NO_x storage process depended strongly on temperature. NO_x storage occurred via both nitrate and nitrite at <200 °C, but solely by nitrate at >250 °C.

When SO₂ (150×10^{-6}) was introduced to the feed gas, a new peak at ca. 1166 cm⁻¹ was detected (Fig. 7(b)), which may have been the chelating bidentate S–O vibration of bulk sulfate [27]. The peak intensity gradually increased with increasing time, and reached a maximum after 0.5 h (Fig. 7(c)). Peaks of nitrate species at ca. 1472 cm⁻¹ and bridging/chelating bidentate nitrate at ca. 1635 cm⁻¹ were still observed, suggesting that the formation of sulfate species had no effect on the NO_x stor-

age process.

The regeneration of Pt/Ba/Al₂O₃-CNTs-SO₂ was performed in 3.5% H₂ at 400 °C, and the results are shown in Fig. 7(d). The sulfate species peak at ca. 1166 cm⁻¹ had largely disappeared, indicating that Pt/Ba/Al₂O₃-CNTs possessed good regeneration ability.

4. Conclusions

Pt/Ba/Al₂O₃-CNTs was prepared and used in NO_x storage and reduction. The catalyst exhibited higher resistance to SO₂ poisoning, and regeneration ability in NO_x reduction, compared with Pt/Ba/Al₂O₃. The superior performance was ascribed to the favorable SO₂ adsorption ability of CNTs, and their resistance against oxidation. This inhibited the oxidation of SO₂ to SO₃ under oxygen-rich conditions. The presence of SO₂ had no obvious effect on the NSR process. Al₂O₃-CNTs is an excellent NSR support in the presence of SO₂, and is an alternative for new sulfur-resistant NSR catalysts.

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Fig. 7. In-situ DRIFTS spectra of Pt/Ba/Al₂O₃-CNTs after exposure to (a) NO (500×10^{-6})-O₂ in Ar for 0.5h at different temperatures; (b) NO(500×10^{-6})-2% O₂-SO₂(150×10^{-6}) in Ar for 0.5 h at different temperatures, (c) NO (500×10^{-6})-2% O₂-SO₂ (150×10^{-6}) in Ar at 200 °C for different time and (d) SO₂ (150×10^{-6}) at 400 °C for 0.5 h, and then reduced by H₂ for different time.

Graphical Abstract

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Alumina-carbon nanotube supports for sulfur-resistant regenerable Pt-based catalysts in NO_x reduction

Lilong Jiang*, Yuling Wang, Xian Liu, Yanning Cao, Kemei Wei
Fuzhou University

Pt/Ba/Al₂O₃-CNTs exhibited high resistance against sulfur poisoning, and good regeneration ability in NO_x removal, in comparison with Pt/Ba/Al₂O₃.

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碳纳米管-氧化铝：高抗硫及再生性能的Pt基催化剂载体 及在NO_x还原反应中的应用

江莉龙*, 王玉玲, 刘 弦, 曹彦宁, 魏可镁
福州大学化肥催化剂国家工程研究中心, 福建福州350002

摘要: 通过乙炔在Al₂O₃上的分解制备碳纳米管-氧化铝(Al₂O₃-CNTs)载体。采用浸渍法, 分别制备了Pt/Ba/Al₂O₃-CNTs和Pt/Ba/Al₂O₃催化剂。利用XRD, SEM, TEM, 低温N₂物理吸附, XPS和in-situ DRIFTS等手段对催化剂的物化性质进行了表征。结果表明, 在SO₂存在下的NO_x还原反应中, Pt/Ba/Al₂O₃-CNTs比Pt/Ba/Al₂O₃具有更高的抗SO₂性能和再生性能。In-situ DRIFTS表明SO₂的存在对NO_x储存还原的途径没有影响。

关键词: 碳纳米管; 氮氧化物净化; 二氧化硫中毒; 再生

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*通讯联系人. 电话: (0591)83731234-8201; 传真: (0591)83707796; 电子信箱: jll@fzu.edu.cn

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