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Preparation of Pt/beta zeolite–Al_2O_3/cordierite monolith for automobile exhaust purification

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Abstract

A two-step route was developed to prepare zeolite beta coatings on structured monolith. Al_2O_3 layer was deposited on cordierite substrate by slurry dip-coating in the first step and beta zeolite layer was then coated on Al_2O_3/cordierite by direct dynamic hydrothermal synthesis in the second step. The as-prepared beta zeolite–Al_2O_3/cordierite was characterized by means of XRD and SEM techniques and the stability of beta coatings was studied. Based on these results, the advantages of the two-step method were discussed. After the introduction of Pt to beta zeolite–Al_2O_3/cordierite, the obtained Pt/beta zeolite–Al_2O_3/cordierite monolith was tested as promising catalyst for the purification of automobile exhaust from real lean-burn engine.

Keywords: Beta zeolite–Al_2O_3/cordierite; Dip-coating; Direct synthesis; Exhaust purification

1. Introduction

Nitrogen oxides (NO*x*), generally referred to NO and NO_2, are major air pollutants that contribute a lot to the formation of photochemical smog and acid rain. Due to the increasingly stringent regulations on NO*x* emissions, various post-treatment methods have been attempted for the elimination of NO*x* emissions from stationary and mobile sources [1]. Selective catalytic reduction of NO*x* by hydrocarbons (HC-SCR, HC: hydrocarbon) is a potential approach to remove NO*x* from automobile exhaust under lean-burn conditions [2]. In the past years, zeolite-based materials have been extensively studied as possible HC-SCR catalysts. Noble metal modified zeolites, *e.g.* Pt/beta zeolite [3–5], exhibit remarkably deNO*x* performance and are regarded promising catalysts for future application. For practical application, catalyst powders are generally fixed on structured substrate to achieve lower pressure drop and shorter diffusion distances, as the case of three-way catalyst (TWC). Therefore, the coating techniques are important issues that show great influence on the performance of monolithic catalyst.

Honeycomb cordierite ($2MgO–2Al_2O_3–5SiO_2$) is the substrate in common use, especially for monolithic automobile catalysts, due to its superior hydrothermal stability and plasticity. There are two general ways to obtain zeolite coatings on cordierite substrate, *i.e.* deposition from slurry of zeolite particles followed by thermal stabilization and direct hydrothermal synthesis [6]. For the former method, it is known that crystalline zeolite materials are difficult to deposit on cordierite and the use of binders are necessary to obtain stable zeolite coatings. The excessive use of binders probably leads to the low utilization ratio of zeolite and corresponding decrease in catalytic activity [7]. The latter method, *i.e.* direct hydrothermal synthesis technique, is a newly developed method to make zeolite coatings on cordierite. ZSM-5/cordierite [8,9] and mordenite/cordierite [10,11] with high zeolite loading and good stability have been obtained. However, the results of direct hydrothermal synthesis are very much related to the synthesis parameters of zeolite powders. As for beta zeolite, it is rather difficult to coat on cordierite substrate by common direct hydrothermal synthesis method [12].

In this work, a two-step method, with the combination of slurry deposition and direct hydrothermal synthesis, to prepare beta zeolite–Al_2O_3/cordierite monolith is reported. The advantages of this two-step method over single slurry deposition method or direct hydrothermal synthesis method are discussed. After the introduction of Pt to beta zeolite–Al_2O_3/cordierite, the as-prepared Pt/beta zeolite–Al_2O_3/cor-

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dierite is tested as promising catalyst monolith for the purification of automobile exhaust from real lean-burn engine.

2. Experimental

2.1. Sample preparation

2.1.1. Preparation of Al_2O_3/cordierite

Cordierite honeycomb monoliths (Corning, 400 cpi, 0.1 mm average wall thickness) were used as substrate. Al_2O_3/cordierite was prepared by traditional dip-coating method. Briefly, γ-alumina (Sinopec, BET surface area 125.2 m^2/g) and H_2O were mixed at the ratio of 1:2 and vigorously stirred for 2 h to form slurry. The pH value of the slurry was carefully adjusted to 5 ± 0.2 by addition of CH_3COOH. The pretreated cordierite was immersed in the slurry for 1 min, taken out and blown with hot air to remove the excess slurry. After dried at 120 °C over night, the monolith was calcined at 600 °C in flowing air for 4 h and labeled as Al_2O_3/cordierite.

2.1.2. Preparation of beta zeolite–Al_2O_3/cordierite

Firstly, Al_2O_3/cordierite was pre-treated in 0.5 M NH_4NO_3 in an ultrasonic bath for 20 min and then dried at 120 °C over night. Then, beta zeolite precursor gel was prepared. Briefly, sodium hydroxide (NaOH), potassium hydroxide (KOH), tetraethylammonium hydroxide (TEAOH), silica powder (SiO_2) and water were mixed under vigorous stirring at the proportion of $28SiO_2$:2NaOH:1KOH:14TPAOH:$400H_2O$. The resulting homogeneous gel was aged at 40 °C for 48 h. Finally, the pre-treated Al_2O_3/cordierite and aged gel were put into teflon lined stainless steel autoclave for crystallization under rotation at 160 °C for 72 h. After crystallization, the monolithic sample was taken out, washed with distilled water ultrasonically, dried at 120 °C over night and then calcined at 600 °C in air for 4 h.

2.1.3. Preparation of beta zeolite/cordierite

For reference, beta zeolite/cordierite was also prepared via two different routes, *i.e.* slurry dip-coating and direct hydrothermal synthesis. For dip-coating, zeolite beta and H_2O were mixed at the ratio of 1:2 and vigorously stirred for 2 h to form slurry. The pH value of the slurry was adjusted to 5 ± 0.2 by addition of CH_3COOH carefully. The pretreated cordierite was immersed in the slurry for 1 min, taken out and blown with hot air to remove the excess slurry. After dried at 120 °C over night, the monolith was calcined at 600 °C in flowing air for 4 h and labeled as beta zeolite/cordierite-dip. For direct hydrothermal synthesis, aged beta zeolite precursor gel and pretreated cordierite were put into teflon lined stainless steel autoclave for crystallization under rotation at 160 °C for 72 h. After crystallization, the monolithic sample was washed, dried, calcined and labeled as beta zeolite/cordierite-hyd. The route is as essential as that employed for the synthesis of beta zeolite–Al_2O_3/cordierite. The only difference is the cordierite is used as substrate for the preparation of beta zeolite/cordierite-hyd while Al_2O_3/cordierite is used as substrate for the preparation of beta zeolite–Al_2O_3/cordierite.

2.1.4. Preparation of Pt/beta zeolite–Al_2O_3/cordierite

Pt/beta zeolite–Al_2O_3/cordierite monolithic catalyst was prepared by impregnating the as-prepared beta zeolite–Al_2O_3/cordierite with aqueous solution of $[Pt(NH_3)_4](NO_3)_2$ at the constant temperature. In a typical preparation process of Pt/beta zeolite–Al_2O_3/cordierite, 50 mL $[Pt(NH_3)_4](NO_3)_2$ aqueous solution (Pt concentration: 4.0 mg/mL) was added to 100 g beta zeolite–Al_2O_3/cordierite to achieve Pt loading of *ca.* 0.2%. The as-prepared Pt/beta zeolite–Al_2O_3/cordierite sample were carefully washed by distilled water, dried at 120 °C over night and calcined at 500 °C in flowing air for 4 h.

2.2. Characterization methods

The X-ray diffraction (XRD) analysis of all samples was carried out on a Rigaku powder diffractometer (D/MAX - RB). The scanning rate was 4° min^{-1} and Cu Kα radiation ($\lambda = 0.1542$ nm) was used.

The surface morphology of samples was examined by scanning electron microscopy (SEM, JEOL JSM 6400) operated at 20 kV. The samples were covered with a thin layer of gold film by sputtering before observation.

The specific surface areas of the samples were determined by nitrogen adsorption at 77 K on Micromeritics ASAP 2010 analyzer.

The loadings of beta zeolite in monoliths prepared by different methods were calculated based on the weight increase before and after preparation.

The stability of beta zeolite–Al_2O_3/cordierite monolith and beta zeolite/cordierite sample was tested by means of ultrasonic vibration on a KQ-800KDE-mode ultrasonic apparatus. After each 15 min, the sample was taken out from ultrasonic, dried and then the weight loss was measured.

2.3. Automobile exhaust purification on Pt/beta zeolite–Al_2O_3/cordierite

The Pt/beta zeolite–Al_2O_3/cordierite was tested as monolithic catalyst for the purification of lean-burn automobile exhaust from real four-valve S. I. engine (1.34 L). The controllable quasi-homogeneous mixture inside the cylinder made by the controllable injection on this engine realized the fast quasi-homogeneous mixture combustion. The air fuel ratio (A/F) was controlled at 20 ± 0.5. The monolithic catalyst (*ca.* 100 g) was fixed inside the vent-pipe of the engine, heated by an electrical heater. The temperature was measured by NiCr–NiAl thermojunction and controlled by relay and temperature controller. The products after catalytic reaction were online analyzed by a five-component exhaust analyzer (MW56-AVL DIGAS 4000 LIGHT).

3. Results and discussion

3.1. Characterization of beta zeolite–Al_2O_3/cordierite

XRD analysis provides us with an easy judgment on the coating results of zeolites on cordierite substrate. Fig. 1

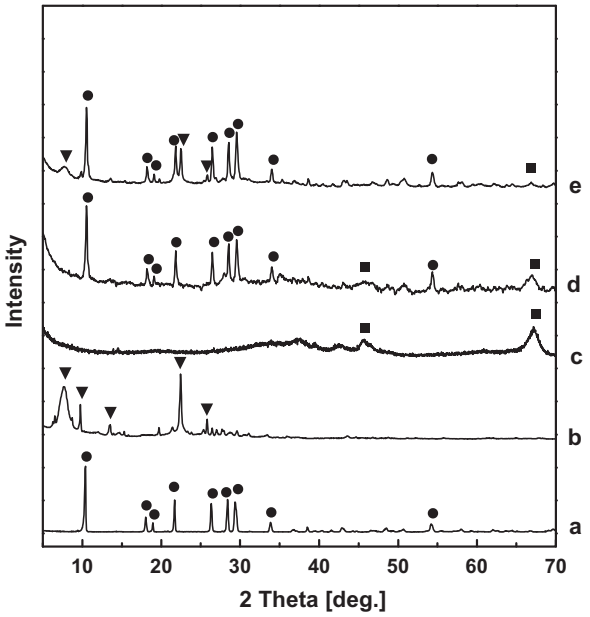

Fig. 1. XRD patterns of cordierite substrate (a), zeolite beta (b), Al_2O_3 (c), Al_2O_3/cordierite (d) and beta zeolite–Al_2O_3/cordierite (e). Circle: cordierite; triangle: beta zeolite; square: Al_2O_3.

compiles the XRD patterns of cordierite substrate, γ-Al_2O_3, beta zeolite, Al_2O_3/cordierite and beta zeolite–Al_2O_3/cordierite. The typical diffraction lines of cordierite, γ-Al_2O_3 and beta zeolite are observed in corresponding samples. While for Al_2O_3/cordierite, diffraction lines corresponding to both γ-Al_2O_3 (*e.g.* at 45.8° and 66.9°) and cordierite (*e.g.* at 10.4° and 29.6°) are observed, indicating the formation of γ-Al_2O_3 coating on cordierite substrate after the first slurry deposition step. For beta zeolite–Al_2O_3/cordierite, diffraction lines corresponding to zeolite beta (*e.g.* at 7.7° and 22.4°) appear. Obviously, beta zeolite coatings are formed on Al_2O_3/cordierite after the second direct hydrothermal synthesis step.

Fig. 2 shows the SEM images of blank cordierite, Al_2O_3/cordierite and beta zeolite–Al_2O_3/cordierite. The irregular ceramic structure with macropores is clearly observed in the image of blank cordierite. After slurry deposition of Al_2O_3, a thin layer is observed to cover the surface of cordierite and the average thickness is estimated to be 50 μm. Meanwhile, a *ca.* 8% weight increase is obtained, indicating the Al_2O_3 loading is *ca.* 8% in Al_2O_3/cordierite. After the second hydrothermal step, characteristic spherical beta zeolite crystals of *ca.* 2 μm are observed to compactly cover the surface of Al_2O_3/cordierite. The loading of beta zeolite is *ca.* 16% based on the weight increase after direct hydrothermal synthesis.

3.2. Advantages of two-step coating method to prepare beta zeolite–Al_2O_3/cordierite

In this work, a two-step coating technique is employed to prepare beta zeolite–Al_2O_3/cordierite, *i.e.* the slurry deposition of Al_2O_3 on cordierite followed by the direct

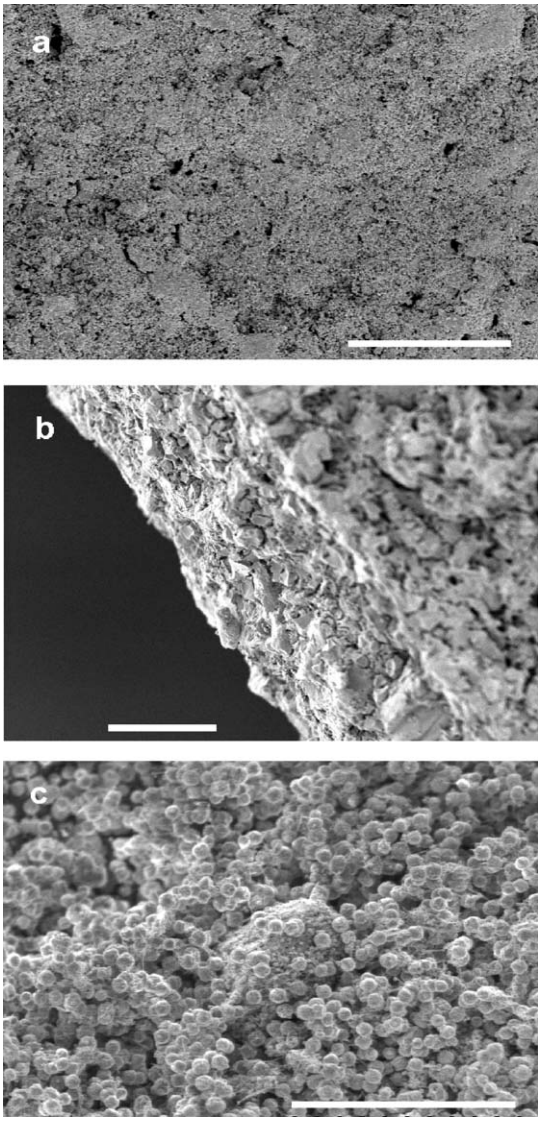

Fig. 2. SEM images of blank cordierite (a), Al_2O_3/cordierite (b) and beta zeolite–Al_2O_3/cordierite (c). Scale bar: 50 μm in each image.

hydrothermal synthesis of zeolite beta on Al_2O_3/cordierite. The final goal of the two-step coating route is obtain beta zeolite coatings on structured ceramic monolith for industrial application. As is known, there are some alternative routes to prepare zeolite beta coatings, *e.g.* slurry deposition of beta zeolite on cordierite and direct hydrothermal synthesis of zeolite beta on cordierite. Here, a comparison of coating results through above-mentioned routes is given as follows.

The weight loading of beta in beta zeolite/cordierite-dip after first dip-coating process (m_{10}) is 6.7% and it increases to 13.9% after the second dip-coating process, as seen in Fig. 3. The weight loading of beta in beta zeolite/cordierite-hyd is 9.1% after first hydrothermal synthesis and it increases to 19.8% after the second hydrothermal synthesis. While for beta zeolite–Al_2O_3/cordierite, beta zeolite weight loading of as high as 16.1% can be obtained after only one hydrothermal synthesis on Al_2O_3/cordierite (Al_2O_3 excluded). The surface area of as-

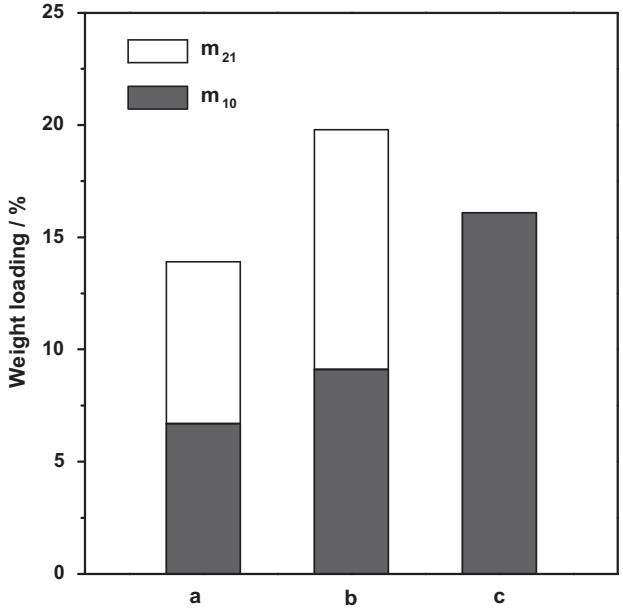

Fig. 3. Coating results of beta zeolite on cordierite substrate by different methods: beta zeolite/cordierite-dip (a), beta zeolite/cordierite-hyd and beta zeolite–Al_2O_3/cordierite (c).

prepared beta zeolite–Al_2O_3/cordierite is *ca.* 37.8 m^2/g and the high surface area is good for the material to be used as monolithic catalyst support.

The stability of zeolite beta coatings on substrate prepared by different methods is evaluated by calculation the weight loss during ultrasonic treatment [13], and the results are shown in Fig. 4. For beta zeolite/cordierite-dip, the weight loading of zeolite beta decreases from 19.8% to 12.7% after 60 min ultrasonic treatment, corresponding to a weight loss of *ca.* 36%. For beta zeolite/cordierite-hyd, the weight loading of zeolite beta decreases from 13.9% to 8.1% after 60 min, corresponding to a more serious weight loss of *ca.* 42%. While a much better stability is achieved on the sample beta zeolite–Al_2O_3/cordierite and the zeolite weight loss of *ca.* 16% is observed (weight loading decrease from 16.1% to 13.6%).

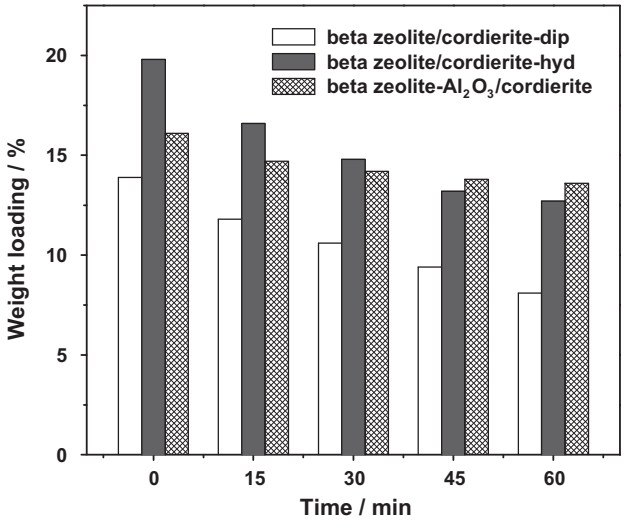

Fig. 4. Stability of beta zeolite coating on cordierite substrate prepared by different methods.

Undoubtedly, zeolite beta coating on Al_2O_3/cordierite with high weight loading and good stability can be prepared via the two-step coating route developed in this study. The origin of the advantages of this two-step coating technique is carefully explained. Slurry dip-coating technique is the most common method to prepare coatings on cordierite and materials with high adhesion, *e.g.* alumina and silica, can be easily coated on cordierite with good stability [14]. For zeolite beta with much lower adhesion, it is difficult to prepare stable zeolite coating on cordierite without the addition of binders [13], as illustrated by the low weight loading and poor stability of beta zeolite coating in beta zeolite/cordierite-dip. For slurry dip-coating with binders, since the binders and zeolites powders are well mixed before dip-coating, the blockage of zeolite by the binders and corresponding decrease in zeolite accessibility are generally unavoidable. Direct hydrothermal synthesis is efficient technique to make zeolite coatings on cordierite substrate without the addition of any binders. However, the coating results are very much dependent on the types of zeolite materials. It is difficult to obtain stable zeolite beta coating through conventional direct hydrothermal synthesis, as proved by the coating results in Fig. 3. The dynamic hydrothermal synthesis has been developed to improve the coating results of beta zeolite coating on cordierite substrate [12]. Moreover, it is much easier to coat zeolite beta on alumina substrate and alumina seems to be better substrate for zeolite beta [15]. So, in this study, we first prepare alumina coating on cordierite substrate and then prepare beta zeolite coating on Al_2O_3/cordierite. Fortunately, stable beta zeolite coating with high weight loading is obtained. In fact, alumina layer acts as a bridge linking beta zeolite coating and cordierite substrate. Since beta zeolite coating is located on the surface of alumina, the blockage of zeolite by alumina is completely avoided.

3.3. Pt/beta zeolite–Al_2O_3/cordierite for the purification of lean-burn automobile exhaust

HC-SCR over Pt/beta zeolite catalyst has been extensively studied in the past and propene is generally used as reducing agent for NO*x* [3–5]. In this study, for practical application, un-burned hydrocarbons and CO are directly used as reducing agent. Under our experimental conditions, the automobile exhaust before purification mainly contains following components: NO*x* = 785 ppm, HC = 152 ppm, CO = 0.09%, O_2 = 5.8%, N_2 = 71%, CO_2 = 11%, H_2O = 11. 5%, SO_2 = 100 ppm. Fig. 5 shows the results of lean-burn automobile exhaust treated by Pt/beta zeolite–Al_2O_3/cordierite monolith. As seen in the figure, the NO*x* conversion increases with increasing temperature in the range from 150 to 300 °C. After reaching to a maximum of *ca.* 63% at 300 °C, the NO*x* conversion gradually decreases below 40% at 450 °C. The HC and CO conversion increases with increasing

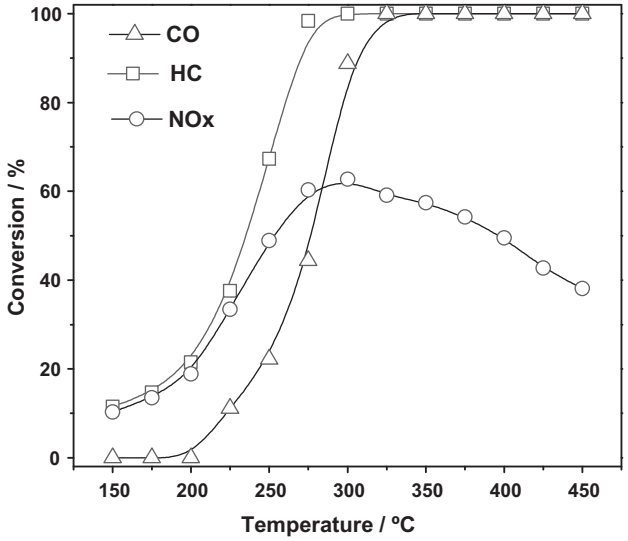

Fig. 5. Temperature-dependent activity of Pt/beta zeolite–Al_2O_3/cordierite monolithic catalysts for the purification of NO*x*, HC and CO from automobile exhaust. *Conditions*: A/F = 20; GHSV = 30,000 h^{-1}; NO*x* = 785 ppm; HC = 152 ppm; CO = 0.09%.

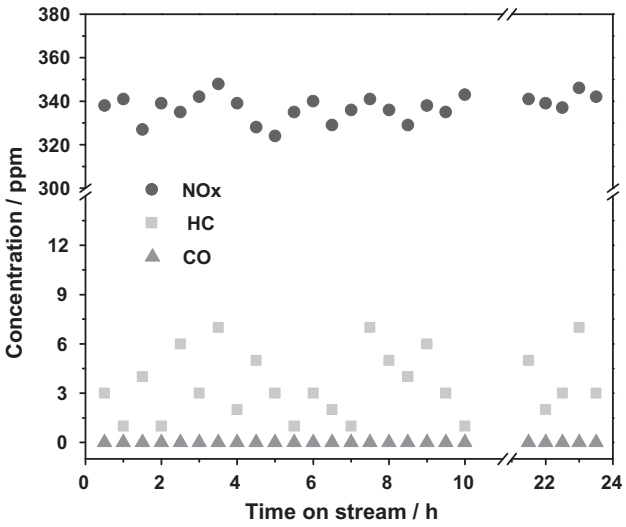

Fig. 6. Time-on-stream behavior of Pt/beta zeolite–Al_2O_3/cordierite for the purification of NO*x*, HC and CO from automobile exhaust at temperature of 325 ± 5 °C. *Conditions*: A/F = 20; GHSV = 30,000 h^{-1}; NO*x* = 785 ppm; HC = 152 ppm; CO = 0.09%.

temperature and an overall conversion can be obtained at 300 and 325 °C, respectively. The completely elimination of HC and CO is achieved at higher temperatures. For real engine test, the purification results are satisfying at 300–400 °C, the temperature window of lean-burn automobile exhaust. Moreover, the good performance of Pt/beta zeolite–Al_2O_3/cordierite is achieved with single active component Pt, further modification by other components may lead to an enhancement in the performance. A comparison between several monolithic Pt catalysts (with similar Pt loading of *ca.* 0.2%) is made and the results are summarized in Table 1. Pt/beta zeolite–Al_2O_3/cordierite (maximal NO*x* conversion: 61.8%) and Pt/beta zeolite/cordierite (maximal NO*x* conversion: 62.7%) show similar deNO*x* activity, higher than Pt/Al_2O_3/cordierite (maximal NO*x* conversion: 49.6%). Obviously, the presence of zeolite beta as support for Pt is essential to achieve high deNO*x* activity. After reaction for 24 h, the deNO*x* activity of Pt/beta zeolite–Al_2O_3/cordierite and Pt/Al_2O_3/cordierite are well preserved, as indicated by the almost unchanged maximal NO*x* conversions. While the deNO*x* activity of Pt/beta zeolite/cordierite declines after 24 h reaction and the maximal NO*x* conversion decreases from 62.7% to 53.6%. Meanwhile, the loss of Pt and the decrease in Pt dispersion are clearly observed, as seen in Table 1. On a whole, good deNO*x* activity and durability are achieved on Pt/beta zeolite–Al_2O_3/cordierite, which demonstrate the potential of this monolithic catalyst for future application. In our opinion, the good performance of Pt/beta zeolite–Al_2O_3/cordierite should be originated from the advantages of two-step coating method for the preparation of beta zeolite–Al_2O_3/cordierite, as discussed in Section 3.2.

Time-on-stream behavior of Pt/beta zeolite–Al_2O_3/cordierite for the purification of NO*x*, HC and CO from automobile exhaust at temperature of 325 ± 5 °C is shown in Fig. 6. As seen in the figure, less than 350 ppm NO*x* and less than 8 ppm HC are detected in the exhaust during 24 h reaction, while no CO is detected. The results are quite exciting for real engine tests.

Table 1
A comparison between several monolithic Pt catalysts for the purification of automobile exhaust.[a]

Catalyst	Pt loading[b]	Pt dispersion[c]	Maximal NO*x* conversion	Reaction temperature
Pt/beta zeolite/cordierite	0.20%	37.6%	61.8%	300 °C
Pt/Al_2O_3/cordierite	0.19%	31.7%	49.6%	320 °C
Pt/beta zeolite–Al_2O_3/cordierite	0.19%	35.9%	62.7%	300 °C
Pt/beta zeolite/cordierite[d]	0.16%	32.1%	53.6%	300 °C
Pt/Al_2O_3/cordierite[d]	0.19%	31.9%	48.9%	320 °C
Pt/beta zeolite–Al_2O_3/cordierite[d]	0.19%	35.3%	63.2%	300 °C

[a] *Reaction conditions*: A/F = 20; GHSV = 30,000 h^{-1}; NO*x* = 785 ppm; HC = 152 ppm; CO = 0.09%.
[b] Determined by ICP.
[c] Determined by H_2 chemisorption, cordierite excluded.
[d] After reaction for 24 h.

4. Conclusion

Monolithic beta zeolite–Al_2O_3/cordierite has been successfully prepared via a two-step coating route. In the first step, Al_2O_3 layer is coated on cordierite substrate by slurry dip-coating and zeolite beta layer is then coated on Al_2O_3/cordierite by dynamic hydrothermal synthesis method in the second step. Zeolite beta layer with high weight loading of *ca.* 16% and good stability can be obtained and the Al_2O_3 layer in the monolith acts as a bridge linking the zeolite beta layer and cordierite substrate. The two-step coating method reported in this study shows great advantages over single coating method, *e.g.* slurry dip-coating and direct hydrothermal synthesis. With the addition of Pt to beta zeolite–Al_2O_3/cordierite, the Pt/beta zeolite–Al_2O_3/cordierite monolith exhibits good performance for the catalytic purification of automobile exhaust from real lean-burn engine. The main pollutant NO*x*, HC and CO can be simultaneously purified at 300–400 °C, the temperature window of lean-burn automobile exhaust.

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