



# Boron nitride wash-coated cordierite monolithic catalyst showing high selectivity and productivity for oxidative dehydrogenation of propane

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## ABSTRACT

Hexagonal boron nitride (*h*-BN) is a burgeoning catalyst for oxidative dehydrogenation of propane (ODHP) with impressive light olefins selectivity, which deserves further study to optimize the heat and mass transfer process with the aim of promoting the potential industrial application since ODHP is a highly exothermic process. Herein, *h*-BN wash-coated cordierite monolithic catalyst was designed through a chemical vapor deposition method using boron acid and urea as precursors, which was used for ODHP reaction and exhibited a ~16.8% conversion of propane and 82.1% selectivity towards propylene, with only 3.7% selectivity of CO and no detected CO<sub>2</sub>, at temperature of 535 °C and a high gas hourly space velocity (GHSV) of 576,000 mL/g<sub>BN</sub>h. The typical structure of *h*-BN/Cordierite with well-developed straight-channel and short diffusive pathway of the thin wash-coated *h*-BN layer guarantee the mass transfer process at high GHSV, achieving a high propylene space time yield of 18.6 g<sub>C<sub>3</sub>H<sub>6</sub></sub> g<sub>BN</sub><sup>-1</sup> h<sup>-1</sup> and improving the selectivity towards propylene and ethylene by suppressing secondary reactions of deep oxidation. Meanwhile, the high GHSV and excellent thermal conductivity of *h*-BN contribute to the heat transfer during reactions.

## 1. Introduction

Propylene is one of the most feedstock in petrochemical industry, employed in the production of a vast series of chemicals, such as polymers, oxygenates and important chemical intermediates [1]. Oxidative dehydrogenation of propane (ODHP) plays significant roles for producing propylene from the abundant shale gas and natural gas resources, with the remarkable characters of less coke formation and non-equilibrium limit in propane conversion [2–4]. Metal oxides and alkaline-earth metal oxychlorides have been widely researched for ODHP reaction with good reactivity, which however still suffer from low selectivity toward propylene because of the over-oxidation of formed propylene to CO<sub>2</sub> (10–60%) [5]. Hexagonal boron nitride (*h*-BN), a non-metallic material with excellent chemical and thermal stability, has been widely researched in heterogenous catalysis [6,7]. Recently, *h*-BN has been demonstrated impressive activity and selectivity for ODHP, which is much higher than that over traditionally used supported vanadium oxide catalysts [8,9]. For example, in our previous work, we found that a higher activity (20.6%) and selectivity (80.2%) were achieved over edge-hydroxylation treated *h*-BN, with a lower selectivity towards CO (7.9%) and CO<sub>2</sub> (0.5%) than the traditional ODHP processes [9]. Furthermore, the excellent chemical and thermal stability of *h*-BN even at the oxidative atmosphere ensure the remarkable catalytic

stability during ODHP reaction [10].

It is noteworthy that the ODHP reaction is highly exothermic and hot spots are easily produced on the catalyst surface, thus causing secondary reaction of deep oxidation [11,12]. Lin et al. found that hotspots in *h*-BN catalyst bed (less than 1 °C) is effectively avoided compared with the widely used VO<sub>x</sub>/γ-Al<sub>2</sub>O<sub>3</sub> catalyst (8 °C) during the ODHP reaction at a similar propane conversion (25%) by computational fluid dynamics calculation method, which primarily benefit from the superior thermal conductivity of *h*-BN [13]. Besides by using the high thermal conductivity catalyst materials, another efficient method to decrease hotspots and suppress over-oxidation is increasing space velocity [14] and decreasing contact time [15]. Hence, it's of great significance to develop a catalyst optimize mass transfer processes during the ODHP reaction. Monolithic catalysts have attracted great interests in recent years [16] due to their significant advantages such as better mass transfer, lower pressure drop and narrow residence time distribution. Owing to these advantages, monolithic catalysts have been proven applicable to many fields including exhaust gas treatment [17,18], destruction of volatile organic compounds [19], selective catalytic reduction of NO<sub>x</sub> [20] and catalytic combustion [21]. Cordierite monolith is the most widely used catalyst support with low thermal expansivity [22], superior thermal stability [23] and good mechanical strength [24].

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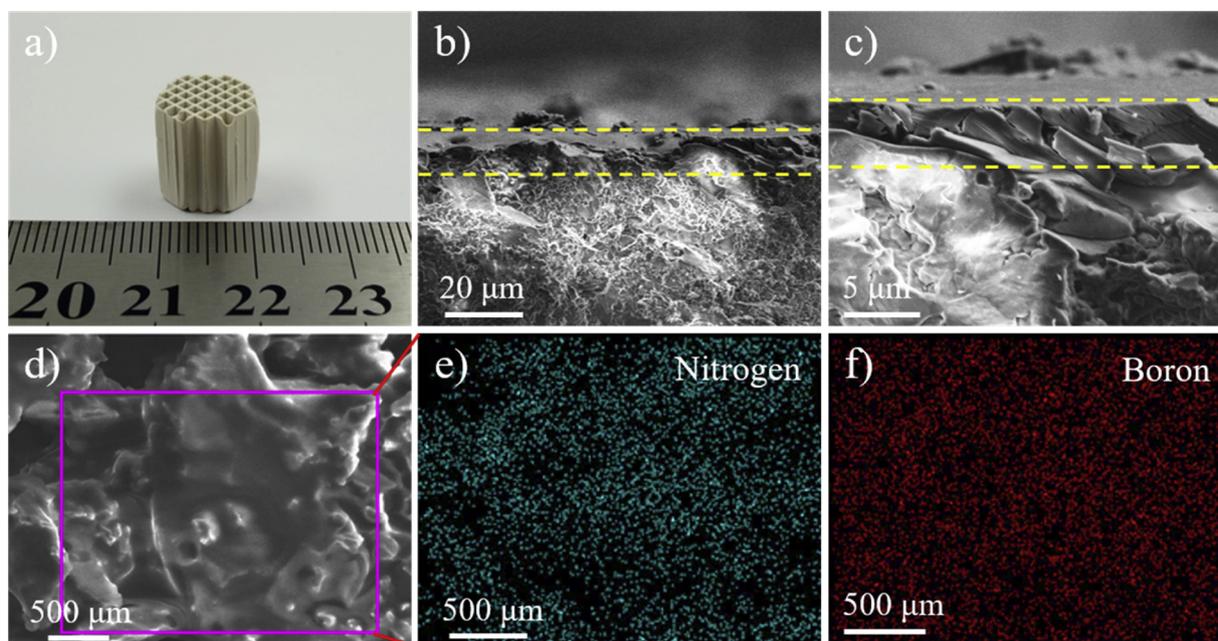


Fig. 1. a) Scheme of monolithic catalyst; b–c) SEM image of cross-section of the cordierite monolithic catalyst wash-coated with *h*-BN; d) SEM image of *h*-BN wash-coat layer; e–f) EDS mapping images of the monolithic catalyst over the zone depicted in (d).

Based on the abovementioned consideration, *h*-BN wash-coated cordierite catalyst was prepared by chemical vapor deposition (CVD) method for ODHP reaction, which exhibits excellent activity, selectivity and stability. The catalytic performance over *h*-BN/Cordierite catalysts with different *h*-BN loading were tested, and the effect of gas hourly space velocity was investigated.

## 2. Experimental

### 2.1. Catalyst preparation

Before using, the cordierite was washed in 4 M HNO<sub>3</sub> at 80 °C for 2 h, followed by extensive washing with ultrapure water, and calcined at 600 °C for 2 h. H<sub>3</sub>BO<sub>3</sub>/Cordierite was obtained after dipping the treated cordierite in boric acid (H<sub>3</sub>BO<sub>3</sub>) solution at 65 °C for 4 h and dried in air at 50 °C. The obtained H<sub>3</sub>BO<sub>3</sub>/Cordierite was placed into an alundum tube, with 2.5 g of urea used as nitrogen precursor, treated at 1000 °C for 1 h at N<sub>2</sub> atmosphere. The catalysts with different *h*-BN loading were prepared by tuning the H<sub>3</sub>BO<sub>3</sub> concentration. Herein, the *h*-BN loading of 1.2%, 2.3%, 3.3%, 4.2% were obtained by tuning H<sub>3</sub>BO<sub>3</sub> concentration of 0.5 M, 1 M, 1.5 M, 2 M. The wash-coat loadings on as-prepared monolith were calculated based on the following formulation:  $W = \frac{m - m_0}{m}$ .

In this formulation, *W* is the wash-coat loading, *m* is the weight of as-prepared monolith and *m*<sub>0</sub> is the weight of blank cordierite before wash-coating [25].

### 2.2. Characterization

The morphology of the catalytic coatings, cross section and elemental chemical analysis was examined with field emission scanning electron microscopy (SEM) using a Hitachi SU8200 instrument.

Fourier transform infrared spectra (FT-IR) was recorded on a Nicolet 6700 FT-IR spectrometer equipped with mercury cadmium telluride (MCT) detector. The powder sample was collected from the surface of the *h*-BN/Cordierite monolithic catalyst. Spectra were averaged over 64 scans in the range 400–4000 cm<sup>-1</sup> with a 4 cm<sup>-1</sup> resolution.

X-ray powder diffraction (XRD) measurements were operated on a PANalytical XPert3 Powder diffractometer using Cu K $\alpha$  radiation

( $\lambda = 0.15406$  nm). The tube voltage was 40 kV, and the current was 40 mA. The sample for test was collected from the cordierite surface.

### 2.3. Adhesion test

To evaluate adhesion of the wash-coated *h*-BN layer, the monolithic catalyst was placed in a sealed round-bottom flask containing petroleum ether and sonicated for 30 min in ultrasonic bath [26,27]. Subsequently, weight changes of the sample were determined after drying the monolith at 30 °C for 1 h. Weight loss was calculated as follows: Weight loss (%) = (W<sub>1</sub> - W<sub>2</sub>)/(W<sub>1</sub> - W) × 100, where *W* is the cordierite weight, W<sub>1</sub> is the wash-coated *h*-BN monolith's weight, and W<sub>2</sub> is the wash-coated *h*-BN monolith's weight after adhesion test.

### 2.4. Activity testing

Catalytic test was run over entire monolith pieces (diameter: 10 mm, length: 10 mm), placed inside a quartz reactor. Before the reaction, each catalyst was subjected to a pretreatment at 500 °C for 3 h under reaction atmosphere (C<sub>3</sub>H<sub>8</sub>: 8 mL/min, O<sub>2</sub>: 12 mL/min, N<sub>2</sub>: 28 mL/min). Reactants and products were analyzed by an online gas chromatograph (Techcomp, GC 7900). A GDX-102 and molecular sieve 5 A column, connected to a TCD were used to analyze the O<sub>2</sub>, N<sub>2</sub>, C<sub>3</sub>H<sub>8</sub>, C<sub>3</sub>H<sub>6</sub>, C<sub>2</sub>H<sub>4</sub>, CH<sub>4</sub>, CO, and CO<sub>2</sub>.

Conversion and product selectivity are calculated using the method presented in our previous work [9]. Typically, conversion was calculated as the number of carbon moles converted divided by the number of carbon moles present in the feed gas. Selectivity was calculated as the number of carbon moles in the product divided by the number of carbon moles reacted. The carbon balance was checked by comparing the number of moles of carbon in the outlet stream to the number of moles of carbon in the feed. Under our typical evaluating conditions, the carbon balance was within  $\pm 5\%$ .

## 3. Results and discussion

*h*-BN/Cordierite catalyst was prepared by a two-step method. H<sub>3</sub>BO<sub>3</sub> was first deposited onto the cordierite by hydrogen bonds interaction through a dip-coating method. The H<sub>3</sub>BO<sub>3</sub> was converted into B<sub>2</sub>O<sub>3</sub> by

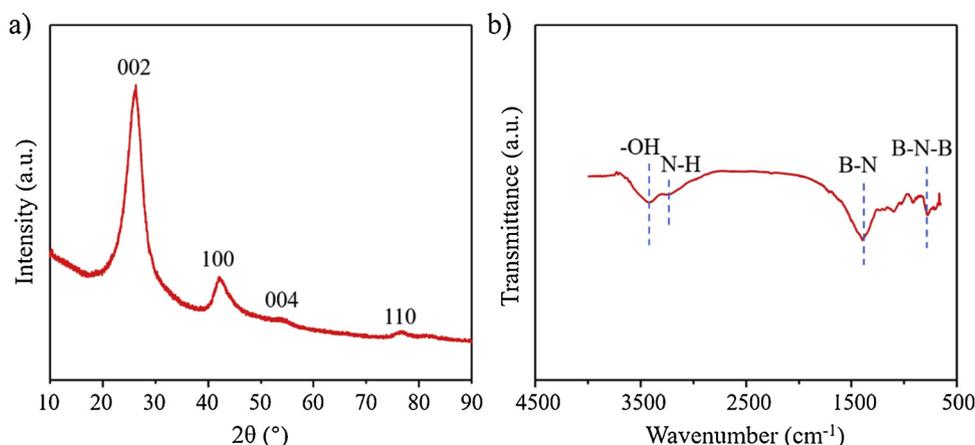


Fig. 2. a) XRD pattern and b) IR spectrum for *h*-BN/Cordierite.

heating and then reacted with  $\text{NH}_3$  derived from decomposition of urea to generate *h*-BN layers [28]. The morphology and textural properties of the catalysts were investigated over a representative *h*-BN/Cordierite catalyst with 4.2% *h*-BN loading. The monolithic catalyst presented a honeycomb-type circular section with a diameter of  $\sim 1.0$  cm, a density of approx. 50 cells/ $\text{cm}^2$ , 0.3 mm of wall thickness and a 55% open frontal area (Fig. 1a). SEM result for the cross-section of the *h*-BN/cordierite clearly showed that the catalytic film is attached to the monolith wall with the thickness of  $\sim 3$   $\mu\text{m}$  (Fig. 1b, c). The monolithic catalyst with a stone-like morphology shown in Fig. 1d owned very small specific surface area of  $\sim 7$   $\text{m}^2/\text{g}$ . The EDS mapping of boron and nitrogen showed a highly and uniformly dispersed *h*-BN over the monolithic catalyst (Fig. 1e–f).

XRD pattern for the surface of *h*-BN/Cordierite was shown in Fig. 2a. The peaks namely (002), (100), (110) were indexed as hexagonal BN (ICDD 00-034-0421). Fig. 2b showed FTIR spectrum for surface layer of the *h*-BN/Cordierite catalyst. A strong absorption band centered at  $1398$   $\text{cm}^{-1}$  is characteristic of an in-plane B–N bond stretching vibration. The sharp peak centered at  $777$   $\text{cm}^{-1}$  is assigned to an out-of-plane B–N–B bending vibration [29]. The bands centered at  $3400$  and  $3200$   $\text{cm}^{-1}$  can be assigned to O–H and N–H stretching bands, respectively [30], which clearly indicate the formation of hydroxyl and amino groups [31]. Combined XRD and IR analysis, we confirm that boron nitride is successfully wash-coated on the cordierite with uniform dispersion by CVD method.

The catalytic results for ODHP over *h*-BN/Cordierite catalyst with 4.2% *h*-BN loading are shown in Fig. 3a, b. The conversion of propane approached 19.7% at  $\sim 510$   $^\circ\text{C}$ , with the products selectivity of  $\text{C}_3\text{H}_6$  (82.2%),  $\text{C}_2\text{H}_4$  (8.2%), CO (9.1%),  $\text{CO}_2$  (0.5%), equivalent to a space

time yield of  $6.97$   $\text{g}_{\text{C}_3\text{H}_6} \text{g}_{\text{BN}}^{-1} \text{h}^{-1}$ . In comparison, a blank experiment (a reactor with cordierite) under the same conditions showed a  $\text{C}_3\text{H}_8$  conversion of only 1.7% at  $\sim 510$   $^\circ\text{C}$  (Fig. 3a). The results indicate that the activity of the monolithic catalyst originates from the *h*-BN wash-coat layer, specifically, the hydrogen abstraction of B–OH groups by molecular oxygen dynamically generates the B–O–O–B intermediates, which further abstracting the hydrogen atoms from alkane, as reported in the previous work [9,32,33].

Considering the loading amount of active phase is a crucial factor for controlling the total reaction rate of a monolithic catalyst, the catalytic properties over *h*-BN/Cordierite with different *h*-BN loading were investigated. With the boron acid concentration increasing from 0.5 M to 1 M, 1.5 M and 2 M, *h*-BN loading amount increased almost linearly from 1.2% to 2.3%, 3.3% and 4.2%. Further, the catalytic performance of *h*-BN/Cordierite with different *h*-BN loading was investigated. As *h*-BN loading increased from 1.2% to 2.3%, 3.3% and 4.2%, the  $\text{C}_3\text{H}_8$  conversion (Fig. 4a) and products distribution (Fig. 4b) almost kept identically. It was speculated that the surface coated *h*-BN plays a catalytic role while the under-layer *h*-BN may be inaccessible, thus, the catalytic performance changed slightly with the increase of *h*-BN loading. Hence, we conclude that the *h*-BN utilization can be improved by coating *h*-BN on the surface of cordierite to form a thin layer.

To illustrate the role of the typical structure of *h*-BN/Cordierite catalyst, a controlled experiment has been performed by using *h*-BN powder as catalyst. *h*-BN powder was diluted with  $\text{SiO}_2$  to ensure the total bed height (10 mm) and the weight of *h*-BN (20 mg) was the same with *h*-BN/Cordierite. As shown in Fig. 5, under the same reaction conditions, the propane conversion over *h*-BN/Cordierite is higher than that over *h*-BN powder. In our previous work for ODH of ethane [34],

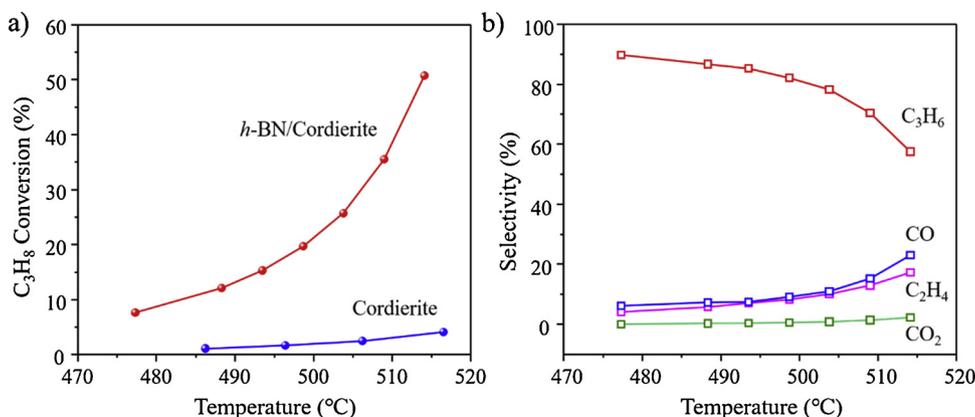


Fig. 3. a) Influence of temperature on the propane conversion over *h*-BN/Cordierite catalyst and cordierite; b) Influence of temperature on the product selectivity over *h*-BN/Cordierite catalyst; Reaction conditions: gas feed, 16.7 vol%  $\text{C}_3\text{H}_8$ , 25.0 vol%  $\text{O}_2$ ,  $\text{N}_2$  balance; flow rate  $48$   $\text{mL min}^{-1}$ .

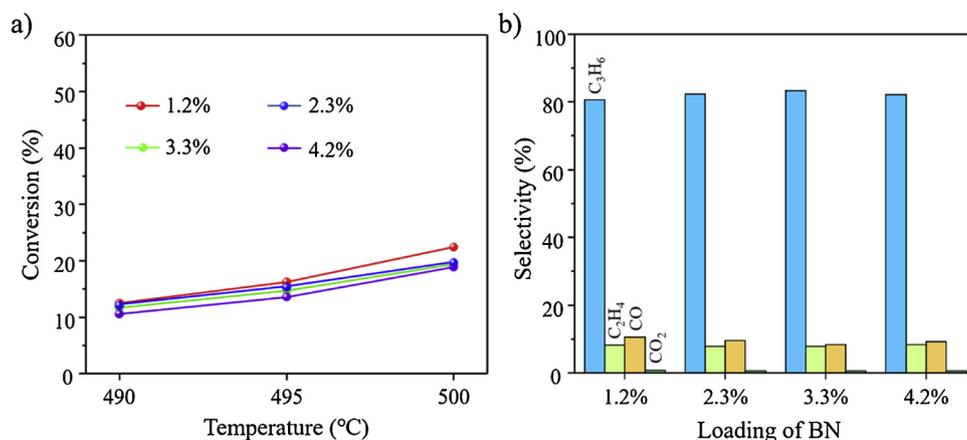


Fig. 4. a) Influence of temperature on the propane conversion over *h*-BN/Cordierite catalyst with different *h*-BN loading; b) The products distribution over *h*-BN/Cordierite with different *h*-BN loading at 500 °C. Reaction conditions: gas feed, 16.7 vol% C<sub>3</sub>H<sub>8</sub>, 25.0 vol% O<sub>2</sub>, N<sub>2</sub> balance; flow rate 48 mL min<sup>-1</sup>.

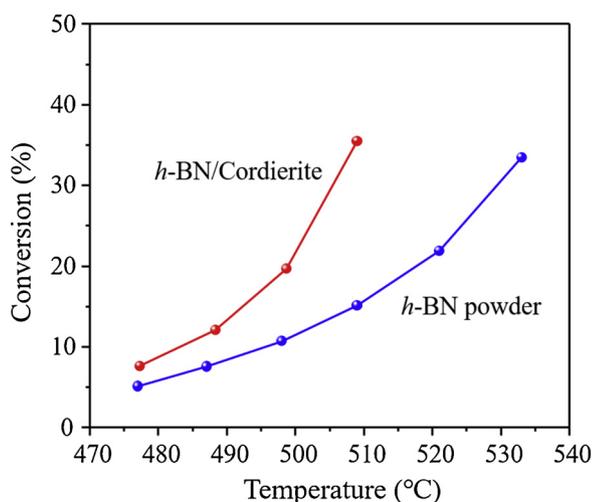


Fig. 5. Comparison of catalytic activity over *h*-BN/Cordierite catalyst and *h*-BN powder catalyst. Reaction conditions: gas feed, 16.7 vol% C<sub>3</sub>H<sub>8</sub>, 25.0 vol% O<sub>2</sub>, N<sub>2</sub> balance; flow rate 48 mL min<sup>-1</sup>.

we proposed that the gas phase free radical reactions play important roles in oxidative of ethane over *h*-BN. We found that the edge B–OH groups may be dehydrogenated by O<sub>2</sub>, generating BO<sup>•</sup> and hydroperoxyl (HO<sub>2</sub><sup>•</sup>) radicals. The BO<sup>•</sup> radical catalyzes hydrogen abstraction of ethane to produce C<sub>2</sub>H<sub>5</sub><sup>•</sup> radical while the HO<sub>2</sub><sup>•</sup> radical may also be involved in the dehydrogenation of ethane in the gas phase. Subsequently, dehydrogenation of C<sub>2</sub>H<sub>5</sub><sup>•</sup> radical in gas phase by O<sub>2</sub> or H<sub>2</sub>O<sub>2</sub> produced the primary product C<sub>2</sub>H<sub>4</sub>. Based on the previous work, we speculate that for ODHP reaction over *h*-BN catalyst, both surface and gas phase reaction play important roles. The well-developed straight-channel structure of *h*-BN/Cordierite provides space for gas phase radical reactions, thus, propane conversion over *h*-BN/Cordierite is higher than that over *h*-BN powder.

The catalyst stability was evaluated through time-on-stream experiment performed at 495 °C and the results are shown in Fig. 6. The conversion of propane stabilized at ~16%, while the selectivity of propylene and ethylene stabilized at 84.5% and 6.5% during the 15 h test. The adherence of the monolithic catalyst layer was evaluated according to a method described in the literature [27], based on the measurement of the weight loss caused by exposure to ultrasounds. The weight loss was only 1.5% even after 120 min tests. Combining with the stability test result, we conclude that the mechanical stability is excellent, which was probably attributed to the outstanding adhesiveness between boron-based compounds and cordierite, no extra binders are

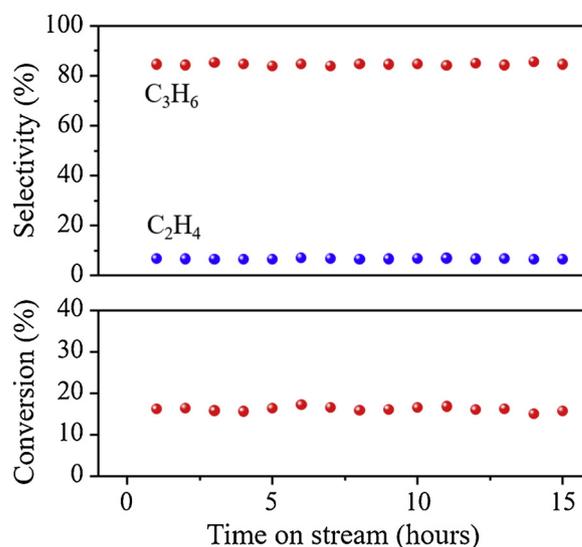
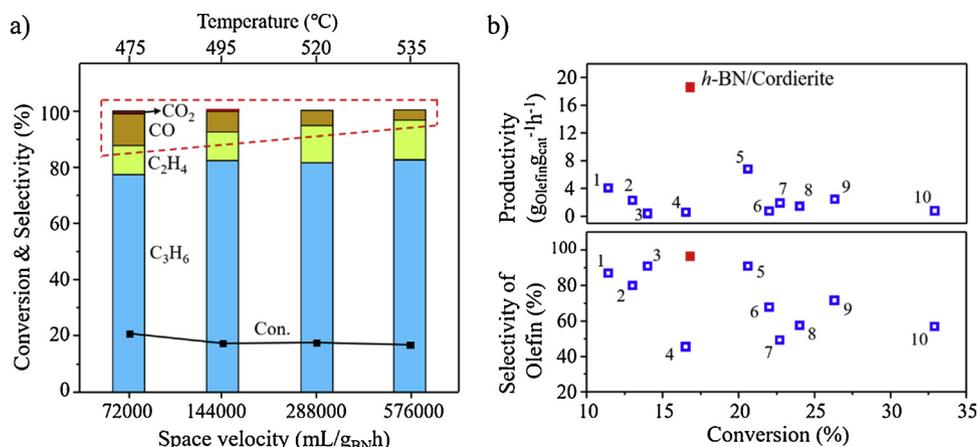


Fig. 6. Catalytic stability tests at 495 °C over *h*-BN/Cordierite. Reaction conditions: gas feed, 16.7 vol% C<sub>3</sub>H<sub>8</sub>, 25.0 vol% O<sub>2</sub>, N<sub>2</sub> balance; flow rate 48 mL min<sup>-1</sup>; temperature, 495 °C.

needed in the wash-coating process. Considering that the cordierite was composed of Al<sub>2</sub>O<sub>3</sub>, MgO and SiO<sub>2</sub>, hydroxyl groups will present on the surface of cordierite. The hydrogen bonds interaction can be formed between the surface –OH of cordierite and B–OH groups of H<sub>3</sub>BO<sub>3</sub>. In addition, CVD process could strengthen the interaction of boron species with cordierite in the form of B–O–Si and B–O–Al, which contributes to the outstanding adhesiveness between *h*-BN layer and cordierite.

The effect of gas hourly space velocity (GHSV) to catalytic performance was evaluated (Fig. 7a). At a GHSV of 72,000 mLg<sub>BN</sub><sup>-1</sup>h<sup>-1</sup>, the C<sub>3</sub>H<sub>8</sub> conversion reached 20.8% at 485 °C, with the products of C<sub>3</sub>H<sub>6</sub> (77.0%), C<sub>2</sub>H<sub>4</sub> (10.5%), CO (11.5%), CO<sub>2</sub> (1.0%). By increasing GHSV to 144,000 mLg<sub>BN</sub><sup>-1</sup>h<sup>-1</sup>, the C<sub>3</sub>H<sub>8</sub> conversion was remained to 17.3% by increasing the reaction temperature to 505 °C, with the selectivity towards C<sub>3</sub>H<sub>8</sub> increased to 82.1%, while that of CO and CO<sub>2</sub> decreased to 7.4% and 0.4%. Moreover, further increasing GHSV to 576,000 mLg<sub>BN</sub><sup>-1</sup>h<sup>-1</sup>, the selectivity towards C<sub>3</sub>H<sub>6</sub> and C<sub>2</sub>H<sub>4</sub> increased to 82.1% and 14.2%, while that of CO decreased to 3.7% and no CO<sub>2</sub> was detected, achieving a high space time yield of 18.6 g<sub>C<sub>3</sub>H<sub>6</sub></sub> g<sub>BN</sub><sup>-1</sup>h<sup>-1</sup>. Both the light olefins selectivity and productivity are higher than the reported *h*-BN and most of metal oxide catalysts. (Fig. 7b) According to the abovementioned results, we conclude that the *h*-BN/Cordierite catalyst can achieve high C<sub>3</sub>H<sub>8</sub> throughput by increasing reaction temperature due to the excellent mass transfer process and



**Fig. 7.** a) Effect of space velocities and reaction temperatures on the propane conversion and products selectivity over the *h*-BN/Cordierite catalyst. **Black square** denotes  $C_3H_8$  conversion, whereas bars represent product selectivity. Reaction conditions: gas feed, 16.7 vol%  $C_3H_8$ , 25.0 vol%  $O_2$ ,  $N_2$  balance; b) Comparison of selectivity and productivity toward light olefins ( $C_3H_6 + C_2H_4$ ) in ODPH as a function of  $C_3H_8$  conversion for several representative catalysts, 1. BNOH, [9] 2. BNNS, [8] 3. V-Mg-O [35], 4.  $V_2O_5/SBA-15$ , [36] 5. VOx-MCM-41 [37], 6. Sol-gel Li/MgO [38], 7. SrVMoOx/ $Al_2O_3$ , [39] 8. CeNbNiOx [40], 9.  $Cr_2O_3/MCF$ , [41] 10. *h*-BN [8].

highly efficient utilization of the active phase. Moreover, at high GHSV, the contact time of reactants is short, which reduces the deep oxidation of reactant. Furthermore, increasing the gas-space velocity contributes to solving the serious issue of radial thermal transmission inside the monolith [14].

For the traditional monolithic catalyst, a secondary support with high specific surface area is usually coated onto the monolith prior to the active phase to enhance the dispersion and adherence of the active components. Considering the high specific surface area and high porosity of the second coating, the internal diffusion process usually needs to be considered. Differently, for *h*-BN/Cordierite catalyst, *h*-BN layer acts as the active component without the high surface area second layer. The total mesopore and micropore volume is negligible ( $0.01 \text{ cm}^3/\text{g}$ ) by  $N_2$ -adsorption with the surface area of  $\sim 7 \text{ m}^2/\text{g}$ . Moreover, it was reported that the molecular diffusion in the wash-coat does not affect the reaction rate when the thickness is lower than  $50 \mu\text{m}$  [14,42]. In our case, the *h*-BN layer thickness for *h*-BN/Cordierite with *h*-BN loading amount of 4.2% is  $\sim 3 \mu\text{m}$ . Thus, we conclude that internal mass transfer is negligible for *h*-BN/Cordierite catalyst.

#### 4. Conclusion

We have shown that a *h*-BN/Cordierite monolithic catalyst exhibits excellent activity, selectivity and stability for the oxidative dehydrogenation of propane. *h*-BN/Cordierite catalysts with different *h*-BN loading exhibit almost the same catalytic performance, indicating the improvement of *h*-BN utilization through coating *h*-BN on the surface of cordierite to form a thin layer. Moreover, a high  $C_3H_8$  throughput was achieved by improving the GHSV, leading to a high selectivity towards olefin while minimizing COx formation. Therefore, the *h*-BN/Cordierite monolithic catalyst with excellent olefins selectivity, productivity and optimized mass and heat transfer performance is attractive for industrial application of oxidative dehydrogenation of propane to olefin production.

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