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Carbon doped BN nanosheets for the oxidative dehydrogenation of ethylbenzene**

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Abstract: Carbon-based catalysts have recently been demonstrated great potential for aerobic oxidative dehydrogenation reaction (ODH). However, its widespread application is retarded by the unavoidable deactivation due to the appearance of coking or combustion under ODH conditions. Here we report the synthesis and characterization of porous structure of BCN nanosheets as well as their application as a novel catalyst for ODH. Such BCN nanosheets consist of hybridized, randomly distributed domains of h-BN and C phases, where C, B, and N were confirmed to covalent bond in the graphene-like layers. Our studies reveal that BCN exhibits both high activity and selectivity in oxidative dehydrogenation of ethylbenzene to styrene, as well as excellent oxidation resistance. The discovery of a simple chemical process to synthesize highly active BCN allows the premise of industrial-scale carbocatalysis to be explored.

In recent years, carbon catalysis has drawn an increasing attention by scientific and engineering communities, especially for the oxidative dehydrogenation of alkane. Carbon materials exhibit important advantages over metal-based catalysts mainly owing to their unique chemical versatility, such as tunable acidity, basicity and electron density.^[1,2] As a result, many extended versions of carbon materials, such as carbon nanotubes (CNTs), ^[3] graphites,^[4] nanodiamonds^[5] and porous carbons,^[6] have been applied to ODH of alkane. In general, these carbon materials, in particular activated carbon catalysts, underwent unavoidable deactivations due to coking or poor oxidation resistance.^[5, 7] Much effort is therefore being expended in attempts to provide oxidation resistance for carbon-based materials at elevated temperatures or in oxygen-exposed environments. One of the approaches to improve oxidation resistance is the use of a chemical inhibitor, usually silicon, boron or phosphorus compounds, acting as a protective layer on the carbon surface.^[8] However, the conversion and selectivity of these materials decrease after the inhibitor deposition, which can be explained in terms of active-site coverage and pore blockage. To seek novel catalysts that can stabilize catalytic dehydrogenation with high selectivity in the presence of oxygen are extremely attractive, but it remains challenge in catalysis.

Hexagonal boron nitride (h-BN) is a newly developed novel ODH catalyst by Hermans and coworkers enabling the selective oxidative dehydrogenation of propane to propene, in spite of dissatisfactory activity as compared to nanocarbons.^[9] Nevertheless, because of high in-plane mechanical strength and good chemical inertness even at elevated temperatures,^[10] h-BN keeps stable up to 1000 °C in air, 1400 °C in vacuum, and

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2800 °C in an inert atmosphere, which is highly desirable in aerobic oxidation reaction.^[9] Furthermore, carbon element can be easily doped into BN-nanosheets over wide compositional ranges, which is a viable way to modify the physico-chemical properties of BN to produce new materials, ternary borocarbonitride (BCN) ceramics. The resultant BCN materials show properties complementary to those of graphene and h-BN, enabling a rich variation in electronic structures, properties and applications.^[11] The most key advantages is that the surface area of h-BN can be enormously improved upon carbon incorporation.^[11] Their large surface-to-volume ratio allows more surface active sites accessible in gas phase catalytic reactions. On the basis of the fact that carbon catalysis has a high catalytic performance in ODH reaction, our strategy to regulate reactivity of h-BN in ODH reaction is to impart carbon catalysis functions in the stable h-BN frameworks, where carbon clusters are stabilized in h-BN matrix by forming solid solutions due to their similar crystal structure. The utilization of BCN materials is thus expected to improve the catalytic performance of h-BN, and at the same time to overcome the deactivation issue of pure carbon materials by confining carbon in h-BN matrix.

Herein, we demonstrated a pyrolysis method to produce porous BCN nanosheets, consisting of hybridized, randomly distributed domains of h-BN and C (such as graphene) (Scheme 1), with controlled surface area and chemical composition for carbon catalysis. In brief, different amounts of glucose were mixed with boric acid and urea. The mixture was put into a tube furnace in an ammonia atmosphere for 8 h at 1250 °C (see methods section for details). The obtained samples were named as BCN-x, where x is the glucose weight in percent of boric acid. This material exhibited enhanced stability and remarkable selectivity towards styrene formation in ODH reactions, here as examplized by the ODH of ethylbenzene.



Scheme 1. The idealized structure of the resultant BCN.

XRD pattern of a series of BCN samples annealed at 1250 °C are shown in Figure S1a. They show two broad reflections corresponding to the (002) and (100) planes respectively.^[12] The pattern is similar to that reported earlier for BCN materials with

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different compositions.^[13] This XRD pattern is typical of materials with a graphite-like structure of h-BN. While increasing the quantity of glucose broadens the peaks,^[14] accompanied by a slight shift of the peak to lower angles. This indicates the success of carbon doping in the h-BN lattice, which induces delamination effect in the 2D materials.

The chemical structure was further characterized with Fourier transform infrared (FT-IR; Figure S1b). The broad strong absorption peaks at 780 cm⁻¹ and 1380 cm⁻¹ correspond to the out-of-plan B–N–B bending vibration and the in-plan B–N transverse stretching vibration, respectively.^[15] The peak at ca.1100 cm⁻¹ is assigned to the B–C vibrations.^[16] The small absorption peak at 1623 cm⁻¹ could be contributed to the formation of sp² C–N bonds.^[17] FT-IR results confirm that the atomic-level BCN hybrid structures are achieved, though the exact composition of the samples has to be studied in more details in the future.

The TEM image (Figure 1a) shows that porous structure and a flake-like morphology are distributed in BCN-800. High-resolution TEM (Figure 1b) allows the observation of an interlayer crystal lattice spacing of 0.33 nm. The nanosheets are composed of near 10 stacked layers and have 3-4 nm overall thickness. Such 2D systems with thicknesses below 10 nm and porous structure are particularly interesting for mediating gas-phase catalysis. The selected-area electron diffraction (inset of Figure 1b) also indicates that the in-planar order of the sample is polycrystalline, which might reflect that the C forms domains or constitutes grain boundaries of BN domains.



Figure 1. (a) TEM image of BCN-800, scale bar, 200 nm. (b) HRTEM image of BCN-800, scale bar, 5 nm; inset: selected electron diffraction of BCN-800.

To further confirm the porous structure of BCN, the BET analysis was performed (Figure S2a). The nitrogen adsorptiondesorption isotherms exhibit a typical characteristic of type I curves with an almost horizontal plateau at low relative pressures, indicating their microporous nature. When carbon content of BCN is higher than BCN-600, the isotherms show a hysteresis, indicating the presence of mesopores. The fairly broad hysteresis loop, which closes upon desorption, indicates the presence of capillary condensation and swelling effects presumably generated N₂ condensation in interstitial voids. All BCN materials show narrow pore size distributions, where 0.4-0.8 nm are predominant (Figure S2b). The formation of porous structure for the samples with higher glucose fraction is most likely due to the carbon combustion during the anaphase of pyrolysis. The BET surface area, micropore pore volume, and total pore volume of the porous BCN are summarized in Table S1 for easy comparison. Such a uniform microporosity and tailored pore environment, and particularly large surface area of as high as 1450 m²/g should allow their innovative applications in catalysis.



Figure 2. Spectroscopic characteristics of BCN. (a) XPS survey spectrum of BCN-800, (b) - (d) XPS spectra of B 1s, N 1s and C 1s, respectively.

Figure 2 is the X-ray photoelectron spectroscopic (XPS) spectra of the BCN product. As illustrated in Figure 2a, the XPS survey spectrum of BCN-800 shows that there are boron, carbon nitrogen and oxygen elements. The high resolution XPS spectra of boron, nitrogen and carbon are shown in Figure 2 b - d, respectively. The B 1s signal can be deconvoluted into three peaks centered at 192.0 eV, 190.3 eV and 189.8 eV, corresponding to the B-O, B-N and B-C bonds, respectively^[18]. The percentage of B-O, B-N and B-C bonds calculated from B1s spectrum is 18.1%, 69.4% and 12.5%, respectively. In the N 1s XPS spectrum, the percentage of N-B bond (397.8 eV) is 71.9%, while the N-C bond (398.8 eV) contributes the rest 28.1%. [18c] According to the intensity and energy of the major peak in the B 1s and N 1s spectra, the main configuration for B and N atoms is the B-N bond, which implies the presence of h-BN domains in our synthesized BCN. Some boron and nitrogen atoms (total around 30%) are bounded with carbon atoms by B-C and N-C bonds, separately. In the high resolution XPS C 1s spectrum, three peaks centered at 284.1 eV, 284.6 eV and 286.0 eV are identified. The main peak located at 284.6 eV is close to the value observed in graphene, which can be reasoned by the presence of graphene domains. The small shoulder peak at the higher binding energy (286.0 eV) can be attributed to a C-N bonding structure,^[17] whereas the peak at lower binding energy (284.1 eV) is caused by the formation of C-B bonds.^[16] The XPS data show that the number of C-C bonds is considerably higher than B-C and C-N bonds, strongly suggesting that C domains embed in h-BN matrix, which form the hybridized BCN atomic layers. Elemental analysis by XPS (Table S2) reveals that the carbon and oxygen content is gradually increasing with increasing amounts of glucose. Thus, XPS analysis clearly supports the incorporation of carbon in the h-BN. The high oxygen content of BCN is probably due to its ultrahigh surface area and high porosity.

Raman spectroscopy has been widely used to characterize the structure order of BN and carbon-based materials. Figure S3a reveals the formation of graphitic carbon with well developed graphene domains, which is in accordance with the XPS results. The samples appear a broad D band at around 1300 cm⁻¹, underlining the presence of disorder carbon in the BCN.^[19] The intensity ratio of D-band and G-band (I_D/I_G) increases from 0.69 (BCN-400) to 0.79 (BCN-1000), which implies that the defect and disorder carbon increase as the incorporated carbon content increases in BCN materials. The increased intensity ratio of I_D/I_G is attributed to the large deformation of sp² hybridized structure on the edge of BCN.



Figure 3. EB dehydrogenation activities of various catalysts. (a) Steady-state activities of BCN and h-BN. (b) Catalytic performance of BCN-800 and graphene samples as a function of time on stream for ODH of EB to ST, Reaction conditions: 30 mg of BCN or 3.8 mg of graphene, 150 nmol/ml EB with N₂ balance, O₂: EB = 4:1, total flow rate =10 ml/min, T = 500 °C.

Prior to catalytic test, the extent of oxidation inhibition effect of the BCN was quantified using TGA (Figure S4), comparing with the activated carbon and graphene nanoplateles. When carrying the analysis in air, the activated carbon and graphene nanoplateles start to combust at about 450 and 600 °C, respectively. While the BCN-800 is stable up to 900 °C, above which the increase in the weight is related to the oxidation of BCN to produce corresponding oxides. Clearly, the BCN sample

shows higher anti-oxidation property than the corresponding carbon materials, making it one of valuable catalysts for aerobic oxidation reaction.

Figure 3a displays the catalytic performances of BCN materials with different carbon content (BCN-400, BCN-600, BCN-800 and BCN-1000) for the ODH of ethylbenzene to styrene, together with h-BN as reference samples. It can be noted that the conversion of EB over h-BN is only 5%, owing to a relatively low specific surface area (180 m²/g) and less active sites. The carbon balance of BCN under the present condition is nearly 99%, indicating the high stability of BCN catalyst.

Long-term stability of BCN-800 was conducted at 500 °C (Figure 3b). After a ca. 9 h induction period, the EB conversion over BCN-800 reached a steady platform, with the highest value of 54% and it was maintained at 50% after 30 h reaction, and the ST selectivity stably approached to 89% during the whole process and the main by-products are benzene, toluene and CO₂. For comparison, one representative commercial graphene nanoplateles with the same carbon contents of BCN-800 were also evaluated. As shown in Figure 3b, a deactivation and lower selectivity of graphene nanoplateles could be clearly detected. The carbon balance and high carbon dioxide yield reveal the deactivation of graphene nanoplateles arose from combustion. This can be explained by the most likely intermediates during the activation of O_2 molecules, $O_2^{2^2}$, O^{2^-} and O^- , which are electrophilic in the graphene nanoplateles and thus ultimately cause over oxidation.^[20] Over each BCN catalysts, there are two potential active sites, carbonyl groups and oxygen-terminated armchair edges of BCN (>B-O-O-N<). As for carbonyl groups, boron element as an electron-deficient promoter tends to inhibit the disassociation of O2 and thus induces the generation of nucleophilic oxygen species intermediates, which have been identified as the prerequisite for a high alkene selectivity.^[21] In contrast, the electron-rich nitrogen atoms in BCN make the electron transfer from the BCN to the adsorbed oxygen species easy and consequently facilitate to increase the reaction rate.[22] As for oxygen-terminated armchair edges of BCN, this is contrary to zero-dimensional single-site catalysts, the onedimensional edge can prevent the overoxidation of the adsorbed species caused by a highly reactive radical and thus can boost the ST selectivity.^[9] In addition, after the reaction the structure of BCN-800 is stable as reflected by the XRD and FTIR examination of the used catalyst (Figure S5a and S5b).

The catalytic performance of BCN catalyst is intriguing, because it is much more active than h-BN. The superior activity can be related to the unique properties of BCN catalysts such as higher surface area, desirable carbon-doping and high anti-oxidation property. Notably, the yield of styrene was found to be gradually increased with the increasing of carbon contents. This suggests that carbon-promoted catalytic activity of BCN for the production of styrene presents a synergistically catalytic effect. Over all BCN catalysts, we obtained Arrhenius activation energies from the ODH reaction rates at different temperature (Figure S6) and the reaction orders for ethylbenzene or O_2 from the linear equation of $\ln R = \min Pi$ (Figure S7, Table 1). These values are comparable to previously reported CNT catalyst.^[5] We thus concluded that the kinetic data agree well with the dual-site Langmuir-Hinshelwood model.

Table 1. Kinetic parameters for ODH of ethylbenzene over BCN-x (723–823 K).

Samples	ODH rate at	Activation	Reaction orders	
	773 K [mmolg ⁻¹ h ⁻¹]	energies [kJ mol ^{⁻1}]	EB	0 ₂
BN	0.15	78.6	1.82	0.57
BCN-400	0.39	71.3	0.68	0.32
BCN-600	0.78	70.4	0.67	0.26
BCN-800	1.52	69.3	0.64	0.17
BCN-1000	1.66	69.7	0.60	0.16

To clarify the real active sites of BCN catalysts, the spent catalysts were characterized by XPS and XRD techniques. The XPS elemental analysis shows that the carbon and boron content are the same for the fresh and used catalysts, as given in Table S2, thus implying the high stability of carbon incorporation in BN lattice and no coke formation during ODH reaction. However, oxygen content was found to increase, yet nitrogen content decreased. This finding means that the oxidation of BCN by O₂ occurs during the reaction condition. The B-O bond of used BCN-800 increases compared to fresh sample (Figure S8 B 1s), signaling oxygen functionalization (as there is no B_2O_3 phase detected in XRD). The new peak at 402.0 eV (Figure S8 N 1s) corresponds to pyridine-N-oxide indicates that the graphitic nitrogen in BCN was oxidized under the ODH reaction condition. The C1s spectrum of BCN-800 shows a new shoulder peak at 288.7 eV (Figure S8 C 1s), which is attributed to the carbonyl groups.^[23] These spectroscopic data confirm that an oxygen-terminated armchair edges of BCN (>B-O-O-N<) and carbonyl groups jointly act as active sites for the ODH. reaction, which is in line with the previous reports by Hermans and Su groups. $^{\left[9,\;24\right] }$

To conclusion, we presented a facile synthesis method to produce porous structure of two-dimension BCN nanosheets, consisting of hybridized h-BN and C domains. The high porosity and extraordinary pore structure of BCN endow more active sites and could facilitate the mass and heat transfer of ODH reaction, which restrains the generation of byproducts. The imparting of carbon catalysis functions in h-BN frameworks leads to high activity and ST selectivity of ODH. BCN nanosheets also show high oxidation resistance capability when compared with other conventional carbon catalysts. Moreover, with abundant oxygen-containing functional groups, the porous BCN nanosheets enrich the family of carbon catalysts and may create a platform for potential applications in sustainable photocatalysis, electrocatalysis, etc.

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Keywords: borocarbonitrides • oxidative dehydrogenation • microporous materials • carbocatalysis

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Porous structure of twodimension BCN nanosheets, consisting of hybridized h-BN and C domains were constructed for aerobic oxidative dehydrogenation reactions. The high porosity, novel pore structure and abundant oxygencontaining functional groups endow the BCN with more active sites.



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