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An efficient nanodiamond-based monolithic foam catalyst prepared by a facile thermal impregnation strategy for direct dehydrogenation of ethylbenzene to styrene

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ABSTRACT

Nanodiamond (ND) has long been recognized as an effective carbocatalyst for synthesizing styrene *via* direct dehydrogenation (DDH). However, the induced drastic pressure drop of its powder form limits its industrial application in heterogeneous catalytic process. In this work, we report a facile hexamethylenetetramine nitrate (HN)-assisted thermal impregnation (HNTI) strategy for fabricating a novel nanodiamond-based monolithic foam (ND/CNT-SiC-ms-HN) catalyst through a two-step approach: One is to soak the carbon nanotube-modified SiC foam (CNT-SiC) with the slurry composed of HN, KCl, LiCl, and dispersed ND, and the other is to heat the slurry-soaked CNT-SiC (ND-HN-KCl-LiCl/CNT-SiC) in N_2 atmosphere at 750 °C. The as-synthesized ND/CNT–SiC-ms-HN monolithic foam features the enriched surface kenotic C=O by promoted ND dispersion and O-doping, abundant stuctural defects, and improved nucleophilicity by N-doping, originating from the promoted ND dispersion by thermal impregnation (TI) in KCl-LiCl molten salt (MS) and the presence of HN in the annealing process. As a result, the ND/CNT–SiC-ms-HN monolithic foam catalyst by HNTI strategy exhibits 1.5 folds higher steady-state styrene rate (5.49 mmol g^{-1} h^{-1}) associated with 98.4% of styrene selectivity compared to the ND-based monolithic foam catalyst (ND/CNT-SiC). Moreover, the ND/CNT–SiC-ms-HN monolithic foam shows excellent long-term stability for the direct dehydrogenation of ethylbenzene to styrene. This work also comes up with a novel way of preparing other highly-dispersed nanocarbons-based monolithic foam catalysts with promising catalytic performance for diverse transformations.

Nanocarbon materials, for example, graphene, carbon dots, fullerene, carbon nanofiber (CNF), nanodiamond (ND), carbon nanotube (CNT), featuring unique structure and chemical properties have attracted extensive attention for catalyzing various important chemical processes in the past decades [1–4]. Among which, the powdered ND first produced in the 1960s in explosions features 5–10 nm diamond nanoparticles. The distinctive sp^3 inner core and sp^2 shell hybridized nanostructure coupled with large surface area, high thermal and mechanical stability and easily prepared character make it an ideal catalyst in diverse industrial application [5–7]. ND has shown great potential in alternating the traditional Fe-based catalysts for styrene production for the sake of clean, safe and eco-friendly requirement over the direct dehydrogenation (DDH) of ethylbenzene process, since the industrial K-Fe catalyst suffer from quick deactivation because of carbon deposition and potassium loss [8,9]. Moreover, the co-feed superheated steam for the purpose of shifting the chemical equilibrium styrene production and eliminating coke deposition results in high energy consumption [10].

Although ND is beyond the shine as a carbon catalyst in the DDH of ethylbenzene reaction, the commercial ND powder produced from the explosion method tend to block the fixed-bed reactors in industrial process, leading to dramatic pressure drop [11]. Meanwhile, because of the existence of surface force and chemical bonding force, individual ND nanoparticles are easy to form an aggregation [12–14]. According to previous reports [15–19], the exposed surface kenotic C=O groups on ND nanosphere are active sites in ethylbenzene dehydrogenation reaction. During this reaction, C=O is converted to C–O–H, and ethylbenzene is activated to styrene. What's more, the aggregation negatively blocks the effective surface sites and significantly depresses the performance in catalytic process. On the other hand, it is still a great challenge in controlling the supra structural properties of ND in the drastic

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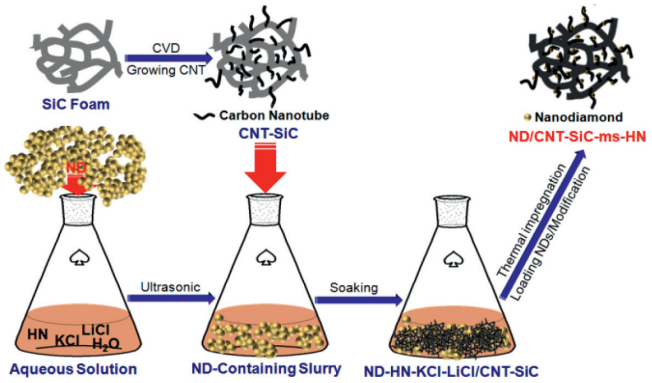
preparation process [20]. On this regard, monolithic catalyst composed of macroporous supports and highly dispersed ND will be an effective protocol for the purpose of overcoming aforementioned disadvantages [21].

Owning to the merits of mechanical stability, chemical stability and good thermal conductivity, silicon carbide (SiC) foam is used as monolithic support diffusely among the macroporous materials to immobilize catalysts for industrial applications [22–24]. However, owning to the chemically inert and low surface area which inhibit the interaction between the supported catalysts and support surface, it is difficult to fix the catalysts firmly on SiC monolith through the tradition preparation method [25]. Thus, it seems reasonable to discover an efficient binding agent to bridge catalysts and SiC support. Su group reported an ingenious method to prepare ND/CNT-SiC monolith [26]. The SiC monolith with in situ grown CNTs coating layer wrapped the commercial ND aggregates by a sonication method and the ND aggregates are homogeneously immobilized within the 3D CNTs-modified SiC foam. Compared with the pristine powder-formed ND, the as-prepared ND/CNT-SiC material exhibited the superior catalytic stability and styrene selectivity in the DDH reaction. However, the incorrigibly obstinate ND aggregates still lowered the use ratio of surface kenotic C=O groups on ND nanoparticles. Typically, chemical bonding force and surface force are main obstacles for the achievement of individual dispersion of ND nanoparticles and the forces can even re-aggregate the de-aggregated ND nanoparticles and the aggregation is much severer at high temperature (reaction temperature for the dehydrogenation reaction is higher than 600 °C). Consequently, to find a facile method for synthesizing loaded ND monolithic foam catalyst on SiC foam with high ND dispersion is urgent.

Molten salt (MS) assisted nanomaterial synthesis process has been proven to be a valid method for the fabrication of mesoporous nanocarbon materials [27–29]. MS is composed of eutectic salts, and it has characteristics of high heat capacity and chemical stability at certain temperature [30–32]. Also, ND nanoparticles tend to aggregate to reduce the total surface energy at high temperature in dry powder [33]. The liquid MS solvent provides a powerful hatch for the reaction of ND and support surface for the lowering in surface energy and it has been proven to be a feasible avenue for the dispersion and de-aggregation of ND nanoparticles at warm atmosphere according to our previous work [34]. The intensified interconnection of ND and support suppress the polymerization of as-separated ND nanoparticles after the washing of MS at room temperature. On the other hand, the reaction of ND and support at high temperature under liquid surrounding intensify the interaction between ND nanoparticles and support which distinctly enhance the catalytic properties in the ethylbenzene DDH reaction. However, it only obtained the powdered carbocatalyst (ND/CN-ms-o), which is hard to be applied in industrial production. In our pervious work, it has been proved that the existence of hexamethylenetetramine nitrate (HN) would realize the O,N-doping and structural defects on the surface of nanocarbons to obtain a higher catalytic activity [35].

Herein, we have prepared the ND/CNT-SiC-ms-HN hybrid monolithic foam by a facile hexamethylenetetramine nitrate (HN)-assisted thermal impregnation through a two-step approach: One is to soak the carbon nanotube-modified SiC foam (CNT-SiC) with the slurry composed of HN, KCl, LiCl, and dispersed ND, and the other is to heat the slurry-soaked CNT-SiC (ND-HN-KCl-LiCl/CNT-SiC) in N_2 atmosphere at 750 °C. In this process, the thermal impregnation method to replace traditional wet impregnation in literature [26] would promote the ND nanoparticles disperse more evenly on the CNT-SiC foam, while the addition of hexamethylenetetramine nitrate (HN) would realize the O,N-doping and structural defects on the surface of nanocarbons [35]. The ND/CNT-SiC-ms-HN hybrid monolithic foam catalyst shows outstanding catalytic performance for the synthesis of styrene, a commercially important chemical, from direct dehydrogenation of ethylbenzene. This work not only reports a monolithic foam carbocatalyst overcoming the shortcomings of powder catalysts with excellent catalytic performance for DDH reaction, but also offers a new direction for fabricating other excellent nanocarbons-based monolithic foam catalysts towards different chemical transformations.



Scheme 1. Schematic illustration for the preparation of ND/CNT-SiC-ms-HN monolithic foam catalyst.

The preparation process for ND/CNT-SiC-ms-HN is illustrated in Scheme 1, which involves three steps: (1) The ND aggregates were added into the aqueous solution composed of HN, KCl, and LiCl to form ND-containing slurry under ultrasonic; (2) The pre-synthesized CNT modified SiC foam (CNT-SiC) by CVD method was soaked into the ND-containing slurry to form the supported ND, HN, KCl, and LiCl precursor on CNT-SiC (ND-HN-KCl-LiCl/CNT-SiC); (3) A thermal impregnation process was performed to prepare the ND/CNT-SiC-ms-HN monolithic foam with high ND dispersion, nitrogen doping, oxygen doping and abundant surface defects through annealing the as-formed ND-HN-KCl-LiCl/CNT-SiC precursor at 750 °C in N_2 atmosphere. For comparison, using the similar process of ND/CNT-SiC-ms-HN to prepare ND/CNT-SiC-ms monolithic foam except for the absence of HN, and also the ND/CNT-SiC monolithic foam was prepared by a traditional wet impregnation method reported in literature [26].

The morphology of the as-made samples was characterized by SEM and TEM. From Fig. 1a, the SEM image of CNT-SiC, SiC surface was entangled by CNT randomly, the diameter of CNT is around 20 nm. Fig. 1b shows that ND/CNT-SiC monolith features 3D CNT networks uniformly covered with the ND clusteres. From Fig. 1c, the highly dispersed NDs were uniformly distributed within the CNT network after the MS-assisted treatment at high temperature. From Fig. 1d and Fig. S1 (Supporting information), ND/CNT-SiC-ms-HN monolith shows both the deposition of ND nanoparticulars and the drastic decompostion of HN does not change the structures of the CNT-SiC foam. It also revealed that the 3D CNT networks can be uniformly covered by de-aggregate ND nanoparticles. ND nanoparticles in ND/CNT-SiC-ms-HN also maintained the original morphology as ND/CNT-SiC-ms under the stroke of HN decomposition implying the strong interaction between ND nanoparticles and CNT-SiC support which is beneficial for the synergistic effect occurring under the dehydrogenation reaction. The TEM images with high magnification were collected in Figs. 1e-h to distinctly clarify the morphology of CNT overlayer and the dispersion of ND nanoparticles. As shown in Fig. 1e, CNT grew randomly, promoting the crosslink between mono CNT and the formation of inner space within the CNT network for the dispersion of ND nanoparticles. Meanwhile, ND nanoparticles are separated around CNT for all three catalysts, implying the insertion of ND nanoparticles into the CNT networks. At the same time, compared to that of ND/CNT-

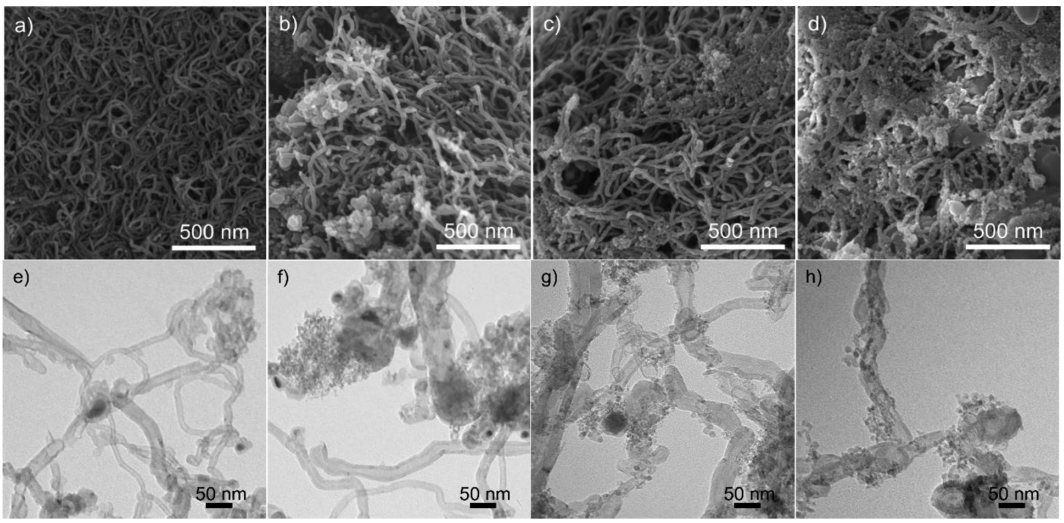


Fig. 1. SEM images of (a) CNT-SiC, (b) ND/CNT-SiC, (c) ND/CNT-SiC-ms and (d) ND/CNT-SiC-ms-HN; TEM images of (e) CNT-SiC, (f) ND/CNT-SiC, (g) ND/CNT-SiC-ms and (h) ND/CNT-SiC-ms-HN.

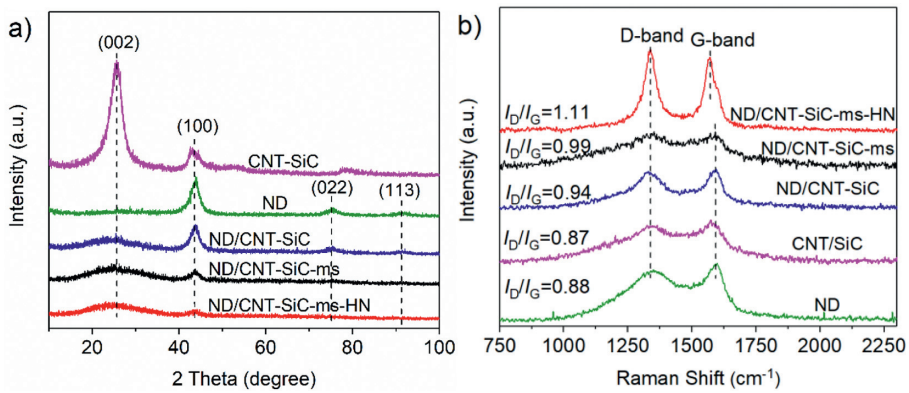


Fig. 2. (a) XRD patterns and (b) Raman spectra of the samples.

SiC (Fig. 1f), the ND nanoparticles were deaggregated in a certain degree under the assistant of molten salts (Fig. 1g). However, ND/CNT-SiC-ms-HN presented the same morphology for ND aggregates, even through HD was drastically decomposed in the preparation process. Overall, CNT kept the original morphology as CNT-SiC after the sequent treatment.

The structures of the as-prepared catalysts were studied by XRD analysis (Fig. 2a). The peaks ascribed to the (002), (100), (022) and (113) crystal faces of ND located at 27.2°, 43.9°, 75.5° and 91.0°, respectively [36,37]. As for CNT-SiC, (002) and (100) crystal faces located at 25.3° and 44° [38]. ND/CNT-SiC sample, a combination of ND with CNT-SiC, presents less-intense diffraction peaks in comparison with ND and CNT, which indicates that ND aggregates was partially de-aggregated and the lack of interaction between ND and CNT. ND/CNT-SiC-ms shows broad and weaker XRD diffraction peaks, indicating highly dispersion of ND aggregates. This may lead to the enhanced interaction between ND and CNT-SiC support. However, we cannot exclude the rearrangement of carbon atom which cannot be clarified in TEM and SEM. Meanwhile, the peak intensity ND/CNT-SiC-ms-HN was further decreased compared with that of ND/CNT-SiC-ms, implying the creation of structural defects and the introduction of nitrogen after the stroke from the explosion of HN at high temperature. Fig. 2b present the Raman spectra of as-prepared catalysts, two broad bands appearing at 1350 cm^{-1} and 1591 cm^{-1} can be denoted as D band (structural defects) and G band (sp^2 carbon), respectively [39,40]. The I_D/I_G commonly used to estimate the extent of disordered graphitic carbon. The I_D/I_G value of ND is 0.88, and that of CNT-SiC is 0.77. While The I_D/I_G value for the mixture, ND/CNT-SiC, is 0.94, which indicates the dispersion of ND nanoparticles. The I_D/I_G value for ND/CNT-SiC-ms is higher than that of ND/CNT-SiC, and this is evidence of more defects in ND/CNT-SiC-ms. As expected, ND/CNT-SiC-ms-HN presented much higher I_D/I_G value in comparison with that for ND/CNT-SiC-ms, since more structural defects appeared under the impact from the decomposition of HN.

The XPS survey spectra show that the samples consisted of C, N and O (Fig. S2a in Supporting information). The C 1s spectra (Fig. S2b in Supporting information) can be resolved into four types of carbon: graphitic structure (C=C) at ~284.6 eV, C–N/C–C at ~286.0 eV, C–O at ~286.6 eV and C=O/C=N at ~288.5 eV [41]. The C=O/C=N concentration of ND/CNT-SiC-ms is much higher than that of ND, CNT-SiC and ND/CNT-SiC, resulting from the high dispersion of NDs. However, after introducing HN in the preparation process, the C=O/C=N content was drastically lost which may be attributed to the gas attack from the decomposition of HN. For the resolved N 1s result, the spectra can be deconvoluted into four peaks occurring at 398.0, 399.3, 400.7 and 402.8 eV, assigned to pyridinic N, pyrrolic N, graphitic N and oxidized N, respectively [42]. From Fig. S2c (Supporting information), enhanced pyrrolic N appeared under the assistant of HN at evaluated temperature. As to oxygen species (Fig. S2d in Supporting information), the peak labeled as C=O is located at 530.8 eV, and the O=C–O is

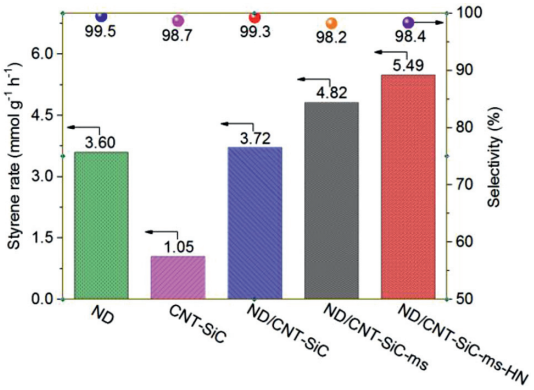


Fig. 3. Catalytic performance over the as-prepared ND, CNT-SiC, ND/CNT-SiC, ND/CNT-SiC-ms and ND/CNT-SiC-ms-HN. Reaction conditions: 30 mg carbocatalyst, 550 °C, 2.8% ethylbenzene in Ar, 10 mL/min flow rate, 20 h for steady-state rate.

located at 532.5 eV. The peak at 533.8 eV can be indexed as C–O in phenol/ether [43]. Quantitatively, the contents of C=O group are 0.4 at%, 0.3 at%, 0.6 at%, 0.7 at% and 0.9 at% for ND, CNT-SiC, ND/CNT-SiC, ND/CNT-SiC-ms and ND/CNT-SiC-ms-HN, respectively. The higher ketonic carbonyl content for ND/CNT-SiC-ms and ND/CNT-SiC-ms-HN originates from the improved dispersion of ND within CNT networks on SiC foam, which is favorable for the DDH reaction since the surface carbonyl groups act as the catalytically active sites for the DDH reaction of ethylbenzene [44,45].

On the basis of the above analysis, a novel nanodiamond-based monolithic foam (ND/CNT-SiC-ms-HN) was prepared by a HNTI strategy, in which the annealing process in the existence of MS can effectively promote the ND dispersion and the addition of HN can realize the N,O-doping and structural defect formation. As a consequence, ND/CNT-SiC-ms-HN monolithic foam possesses more exposed actvie sites (kenotic C=O groups and structural defects) with improved nucleophilicity by N-doping, which allows it to be an excellent monolithic foam carbocatalyst for direct dehydrogenation of ethylbenzene. The catalytic performance was measured by using the same weight of nanocarbons. From Fig. 3, the ND/CNT-SiC monolithic foam, prepared by the previously reported method in literature [26], even shows higher activity with 3.72 mmol g^{-1} h^{-1} of styrene rate and 99.3% of styrene selectivity compared to the powder-formed ND catalyst, although the CNT/SiC foam only shows 1.05 mmol g^{-1} h^{-1} of low DDH activity, suggesting the promoting effect of the improved mass transfer by the preparation. More significantly, the ND/CNT-SiC-ms-HN monolithic foam catalyst exhibits 1.5 folds of steady-state styrene rate (5.49 mmol g^{-1} h^{-1}) with 98.4% of selectivity compared to the ND/CNT-SiC foam catalyst prepared by the previously reported traditional wet-impregnation (WI) method, ascribed to the enriched surface kenotic C=O by promoted ND dispersion and O-doping, abundant structural defects, and improved nucleophilicity by N-doping, originating from the promoted ND dispersion by thermal impregnation in KCl-LiCl molten salt and the presence of HN in the annealing process. Moreover, even if the HN wasn't added, the as-prepared ND/CNT-SiC-ms foam catalyst also shows 1.3 times of steady-state styrene rate compared to ND/CNT-SiC catalyst, suggesting the promoting catalysis of the improved ND dispersion by thermal impregnation. Moreover, the developed ND/CNT-SiC-ms-HN monolithic foam catalyst exhibits excellent stability (Fig. S2 in Supporting information), and it could be a practical carbocatalyst for styrene production *via* ethylbenzene DDH reaction.

In summary, a novel ND-based monolithic foam catalyst was successfully fabricated by a facile hexamethylenetetramine nitrate-assisted thermal impregnation strategy, generating an unexceptionable structured catalyst for styrene production through the ethylbenzene DDH process, ascribed to the enriched surface kenotic C=O by promoted ND dispersion and O-doping, abundant structural defects, and improved nucleophilicity by N-doping led by the promoted ND dispersion by thermal impregnation in KCl-LiCl molten salt and the presence of HN in the annealing process. As a result, the developed ND/CNT–SiC-ms-HN monolithic foam catalyst shows 5.49 mmol g^{-1} h^{-1} much superior steady-state styrene rate with high selectivity to referenced ND/CNT-SiC monolithic foam catalyst previously reported traditional wet-impregnation method similar to that for ND/CNT–SiC-ms-HN except for the absence of HN, KCl and LiCl. The developed monolithic foam catalyst displays a great potential as a metal free catalyst for industrial production of styrene. In addition, this work also paves a new way towards constructing other highly-dispersed nanocarbons-based monolithic foam catalysts with excellent catalytic properties for other reactions.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2022.107808.

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