



## Short Communication

## Hydrolytic dehydrogenation of amine-boranes catalyzed by graphene supported rhodium–nickel nanoparticles

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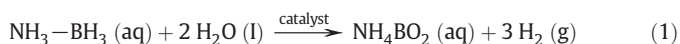
## ABSTRACT

Well dispersed rhodium–nickel (RhNi) nanoparticles (NPs) supported on graphene have been synthesized through an in situ co-reduction procedure under ambient condition, which shows high catalytic activity and durability for hydrolysis of ammonia borane and methylamine borane.

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## 1. Introduction

Ammonia borane ( $\text{H}_3\text{N}-\text{BH}_3$ , AB) complex is considered to be the most promising candidate for portable hydrogen application among all other practical hydrogen storage materials due to its high hydrogen content (19.6% wt), high stability, and environmental benignity [1]. As the derivative of AB, methylamine borane ( $\text{CH}_3\text{NH}_2-\text{BH}_3$ , MeAB) has also been studied due to its high gravimetric hydrogen content (11.1 wt.%). The release of hydrogen from AB and MeAB could be obtained via solid phase thermolysis [2], catalytic dehydrogenation in non-aqueous solvents [3], and hydrolysis [4]. With appropriate catalyst, hydrolysis of AB or MeAB could release as much as 3 mol of hydrogen per mol of AB or MeAB, which appears to be the most convenient one for portable hydrogen storage applications according to Eqs. (1) and (2) [5]. So far, many catalyst systems have been tested for hydrogen generation from the hydrolysis of AB and MeAB; however, the optimal compromise between costs, efficiency and recyclability still remains considerable challenges.



The catalytic performance of the metal NPs is highly dependent on the dispersion of the active metals [6], and one of the most critical problems is the aggregation of NPs into bulk metal during the catalytic process [7]. To solve this problem, appropriate supports have been designed for restraining the agglomeration of the metal NPs [8]. Up to now, various types of supported materials have been employed to uniformly disperse Rh NPs, such as  $\text{TiO}_2$  [9a], zeolite framework [9b], and  $\gamma\text{-Al}_2\text{O}_3$  [9c], and their catalytic activity toward hydrolysis of AB has also been studied. Graphene, a single-atom-layer carbon material holding fascinating properties such as high specific surface area, superior electrical conductivity, high chemical stability, has been widely used as an ideal substrate for growing and anchoring of metal NPs [10]. However, the reports about Rh NPs supported on graphene and their catalytic activity toward hydrolysis of AB, to the best of our knowledge, are rare.

Herein, we report a facile in situ synthesis of graphene supported RhNi NPs using MeAB as reductant in a one-step co-reduction route at room temperature under ambient atmosphere. Their catalytic activities toward hydrolysis of AB and MeAB have also been tested.

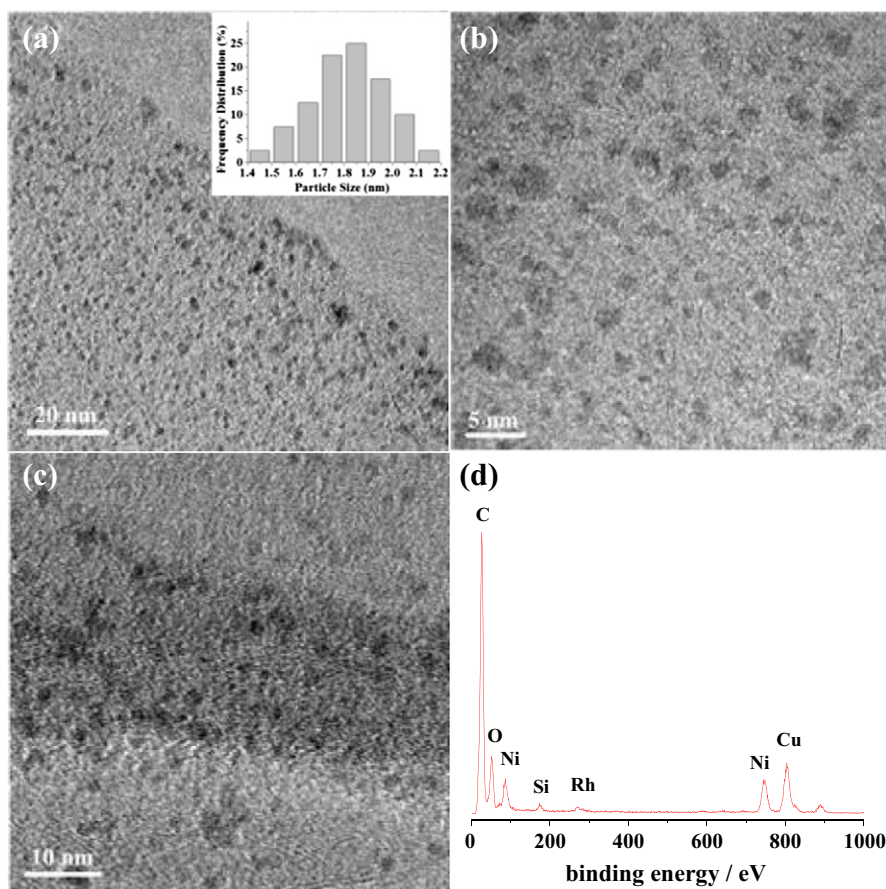
## 2. Experimental

## 2.1. Chemicals and materials

Ammonia borane ( $\text{NH}_3-\text{BH}_3$ , AB, Aldrich, 90%), sodium borohydride (Sinopharm Chemical Reagent Co., Ltd,  $\geq 96\%$ ), rhodium (III) chloride hydrate ( $\text{RhCl}_3 \cdot n\text{H}_2\text{O}$ , TCI Shanghai Co., Ltd. 98%), nickel chloride

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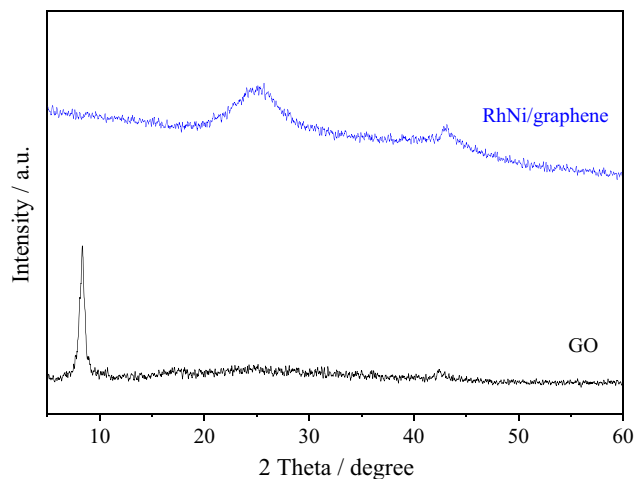
**Fig. 1.** (a)–(b). TEM image of the RhNi/graphene with different magnifications. (a inset) Particle size distributions of RhNi/graphene. (c). TEM image of the RhNi/graphene NPs after the fifth cycle. (d). EDX of RhNi/graphene.

hexahydrate ( $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ , Sinopharm Chemical Reagent Co., Ltd,  $\geq 99\%$ ), methylamine hydrochloride ( $\text{CH}_3\text{NH}_2 \text{HCl}$ , Sinopharm Chemical Reagent Co., Ltd,  $\geq 96\%$ ), potassium permanganate ( $\text{KMnO}_4$ , Shanghai Chemic Co., Ltd,  $\geq 99.5\%$ ), hydrogen peroxide ( $\text{H}_2\text{O}_2$ , Sinopharm Chemical Reagent Co., Ltd,  $\geq 30\%$ ), phosphoric acid ( $\text{H}_3\text{PO}_4$ , Sinopharm Chemical Reagent Co., Ltd, AR), sulfuric acid ( $\text{H}_2\text{SO}_4$ , Sinopharm Chemical Reagent Co., Ltd, 95–98%), tetrahydrofuran ( $\text{C}_4\text{H}_8\text{O}$ , Sinopharm Chemical Reagent Co., Ltd,  $\geq 99\%$ ), dimethyl ether anhydrous ( $\text{C}_4\text{H}_{10}\text{O}$ , Sinopharm Chemical Reagent Co., Ltd,  $\geq 99.7\%$ ), and graphite power

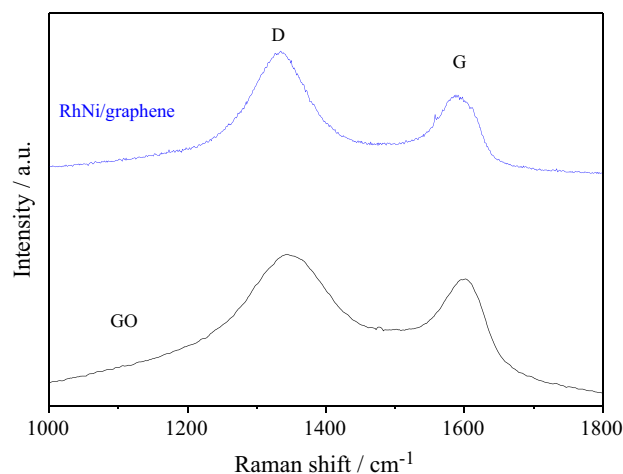
(Sinopharm Chemical Reagent Co., Ltd,  $\geq 99.85\%$ ). All chemicals were used as obtained. We use ordinary distilled water as the reaction solvent.

## 2.2. Graphene oxide (GO) preparation

GO was made by a modified Hummers method [11]. In an improved synthesis of graphene oxide, a 9:1 mixture of concentrated  $\text{H}_2\text{SO}_4/\text{H}_3\text{PO}_4$  (360:40 mL) was added to a mixture of graphite flakes (3.0 g)



**Fig. 2.** XRD patterns of GO and RhNi/graphene.



**Fig. 3.** Raman spectra of GO and RhNi/graphene.

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