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Nickel–platinum Nanoparticles Immobilized on Graphitic Carbon Nitride as Highly Efficient Catalyst for Hydrogen Release from Hydrous Hydrazine

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Lixin Xu^a, Na Liu^a, Bing Hong^a, Ping Cui^a, Dang-guo Cheng^b, Fengqiu Chen^b, Yue An^b and Chao Wan^{a,*}

Here we demonstrate that the combination of NiPt alloy nanoparticles with a graphitic carbon nitride (g-C₃N₄) support facilitates H₂ production from hydrous hydrazine in an alkaline solution under moderate conditions. Of all the heterogeneous catalysts tested, Ni₃₇Pt₆₃/g-C₃N₄ shows superior catalytic performance with a maximum initial turnover frequency (TOF) of 570 h⁻¹ at 323 K.

In light of the upcoming energy crisis and the increasingly severity of environmental pollution it is desirable to use sustainable, renewable and clean energies¹. Hydrogen, whose only byproduct is water, is an environmentally attractive and ideal energy carrier, especially for electricity generation in low-temperature fuel cells²⁻⁵. However, despite the numerous studies conducted over several decades, the hydrogen economy has not been fulfilled due to the challenges in safely and efficiently storing hydrogen⁶⁻⁹. Chemical hydrogen storage is a promising solution because it can be conveniently transported and has a considerable hydrogen content¹⁰⁻¹⁵. Hydrous hydrazine, such as hydrazine monohydrate (H₂NNH₂•H₂O), is considered a promising candidate for storing hydrogen. It is advantageous due to its high hydrogen storage capacity (8.0 wt%), easy recharging as a liquid (the current infrastructure for liquid fuel cells can be utilized), and its environment-friendliness, i.e. the only byproduct is nitrogen via a complete decomposition reaction: H₂NNH₂→N₂(g)+2H₂(g)¹⁶⁻²¹. Nevertheless, to efficiently liberate hydrogen, the undesired reaction 3H₂NNH₂→4NH₃(g)+N₂(g) must be avoided²²⁻²⁷. To solve this problem, one must develop highly efficient, selective, and economic catalysts for producing hydrogen from hydrous hydrazine in practical applications.

For implementing this target, much attention has been devoted to synthesize metal catalysts for improving the hydrogen release from H₂NNH₂•H₂O, among which noble metals, e.g., Pt, Pd, Rh, or their bimetallic alloy catalysts²⁸⁻³⁴ have proved to be efficient. In light of this, many studies have been devoted to developing monometallic and polymetallic nanoparticles (NPs), where materials such as Al₂O₃, CeO₂, graphene oxide, carbon black, etc. act as the support³⁵⁻⁴⁴ and metals such as Pt⁴⁵⁻⁵⁰, Pd^{33,51}, and other elements^{19-21,52,53} are the active ingredients. In particular, binary NiPt alloy NPs are efficient catalysts for hydrous hydrazine decomposition in alkaline solutions. The dispersion of the metal NPs throughout the solution significantly influences the performance of the catalysts⁴⁵⁻⁴⁹; therefore, to improve their activity one must reduce the clustering of NPs via the use of proper supports.

Recently, graphitic carbon nitride (g-C₃N₄) has received a great deal of attention as a metal-free candidate to supplement traditional carbon materials due to its many appealing properties: low cost, semiconductivity, high chemical and physical stability, basicity, energy-storage capacity, and nontoxicity^{54,55}. g-C₃N₄ has been comprehensively investigated for use in gas storage, fuel cells, photocatalysis, and heterogeneous catalysis⁵⁴⁻⁵⁷. It has emerged as one of the more promising supports for metal NPs because the nitrogen-rich structure of its amine-bridged heptazine units provides many adsorption sites for grafting metal ions on its surface⁵⁸. Furthermore, to the best of our knowledge, no NPs have yet been immobilized on g-C₃N₄ supports for the catalyzing H₂NNH₂•H₂O dehydrogenation. Here we present the first instance of NiPt alloy nanoparticles (NPs) supported on g-C₃N₄ and show their excellent catalytic performance in the dehydrogenation of H₂NNH₂•H₂O under moderate conditions.

g-C₃N₄ was prepared via thermal condensation of melamine powder⁵⁴. Melamine powder (5 g) was put in a covered alumina crucible, heated to 823 K at a rate of 5 K•min⁻¹ and maintained at 823 K for 4 h in a nitrogen flow (30 mL•min⁻¹). The final yellow product was collected and ground into powder with an agate mortar.

For the preparation of NiPt/g-C₃N₄ catalysts with variable Ni/Pt ratios (1:0, 1:4, 2:3, 3:2, 4:1, 0:1), 0.2 g of g-C₃N₄ was impregnated by

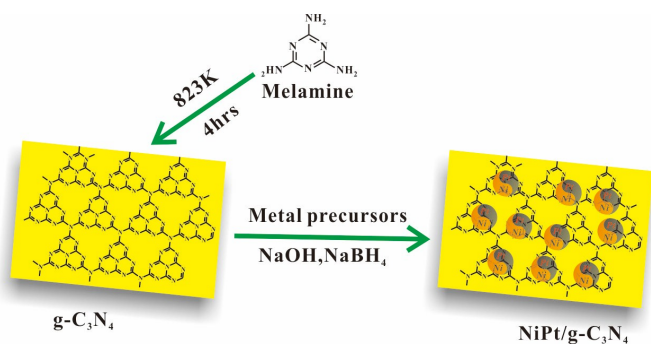
^a College of Chemistry and Chemical Engineering, Anhui University of Technology, 59 Hudong Road, Ma'anshan 243002, China

^b College of Chemical and Biological Engineering, Zhejiang University, 38 Zheda Road, Hangzhou 310027, China

E-mail: wanchao1219@hotmail.com

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Scheme 1 Schematic illustration of the synthesis of the NiPt/g-C₃N₄ catalysts.

a mixed solution containing 0.4 mmol of metal ions with variable molar ratios of Ni²⁺ and Pt⁴⁺, and the solution was diluted with distilled water to 20 mL. After stirring for 24 h at 298 K, a solution containing sodium borohydride (NaBH₄) and sodium hydroxide (NaOH) was added to the above-mentioned mixture while stirring vigorously at 273 K. The catalysts were extracted by centrifugation, washed with ethanol, and dried in vacuum at 373 K for 12 h.

The synthesis of the NiPt/g-C₃N₄ is displayed in Scheme 1. The NiPt/g-C₃N₄ catalysts were reproducibly prepared by this simple impregnation method, followed by reduction by the NaBH₄ and NaOH solution at 273 K⁴⁹. After centrifugation and washing with copious amounts of water, NiPt/g-C₃N₄ was obtained, characterized using various techniques, and was used for the dehydrogenation of hydrous hydrazine. The molar ratio of the NiPt NPs in the catalysts was modified by tuning the initial composition of NiCl₂ and H₂PtCl₆, and determined using inductively coupled plasma atomic emission spectroscopy (ICP-AES). As shown in Table S1 (electronic supporting

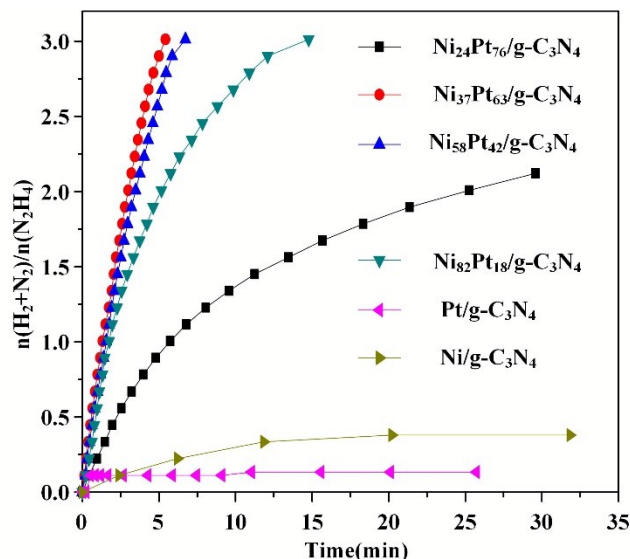


Fig. 1 Time course plots for the decomposition of hydrous hydrazine over NiPt/g-C₃N₄ with NaOH (0.5 M) at 323 K (catalyst=0.100 g; N₂H₄•H₂O=0.1 mL).

information; ESI), the Ni/g-C₃N₄, Ni₂₄Pt₇₆/g-C₃N₄, Ni₃₇Pt₆₃/g-C₃N₄, Ni₅₈Pt₄₂/g-C₃N₄, Ni₈₂Pt₁₈/g-C₃N₄, and Pt/g-C₃N₄ catalysts were synthesized using metal precursors at Ni/Pt molar ratios of 1:0, 1:4, 2:3, 3:2, 4:1, and 0:1, respectively. The catalytic reactions were performed in alkaline solutions of hydrazine at 323 K, as shown in Fig. 1 and Table S2 (ESI). The performances of the catalysts were distinctly improved when Pt was alloyed to Ni. Ni₃₇Pt₆₃/g-C₃N₄ showed the best catalytic performance and a maximum turnover frequency (TOF) of 570 h⁻¹ at 323 K, which is much higher than most of literature values for NiPt catalysts (Table 1)⁴⁵⁻⁴⁹. The 100% hydrogen selectivity of the decomposition reaction of hydrous hydrazine over Ni₃₇Pt₆₃/g-C₃N₄ was verified using mass spectrometry (Fig. S1, ESI). Pt/g-C₃N₄ was nearly catalytically inactive, but catalytic activity increased by alloying and increasing the composition of Ni, which highlights the existence of synergistic effects on the molecular level from alloying NiPt NPs.

The dehydrogenation of hydrous hydrazine catalyzed by the Ni₃₇Pt₆₃/g-C₃N₄ was conducted at different temperatures (298–343 K) to acquire the activation energy (E_a) of this reaction. The values of the rate constant (k) were acquired from the slope of the linear part of the graph of dehydrogenation reaction versus time, at different temperatures (T) in Fig. 2(a). The Arrhenius plot (ln k vs. 1/T) for this catalyst is displayed in Fig. 2(b). E_a is determined to be 36.6 kJ/mol, which is smaller than most of literature value for this reaction over NiPt catalysts (Table 1). Furthermore, the recyclability of the Ni₃₇Pt₆₃/g-C₃N₄ catalyst was investigated during the decomposition of hydrazine hydrate at 323 K (Fig. S2, ESI). After the four times cycle run, the catalytic activity of catalyst decreased slightly and hydrogen selectivity remained stable. As shown in Fig. S3, the reason for the decrease in the dehydrogenation performance can be attributed to agglomeration of the particles, which is consistent with the literature findings^{47,49}.

The X-ray diffraction (XRD) patterns of the g-C₃N₄, Ni/g-C₃N₄, Pt/g-C₃N₄, Ni₂₄Pt₇₆/g-C₃N₄, Ni₃₇Pt₆₃/g-C₃N₄, Ni₅₈Pt₄₂/g-C₃N₄ and

Table 1 Comparison of the decomposition of hydrous hydrazine over different catalysts in this study and previous reports.

Catalyst	T (K)	TOF (h ⁻¹)	E _a (kJ/mol)	Reference
Ni ₃₇ Pt ₆₃ /g-C ₃ N ₄	323	570	36.6	This work
Ni _{0.99} Pt _{0.01}	323	12	49.95	59
Ni _{0.9} Pt _{0.1} /Ce ₂ O ₃	298	28.1	42.3	36
Ni ₈₄ Pt ₁₆ /graphene	323	415	40	44
Ni ₈₈ Pt ₁₂ @MIL-101	323	375.1	51.29	43
Ni ₃ Pt ₇ /graphene	323	416	49.36	45
Ni ₆₀ Pt ₄₀ /CeO ₂	303	293	58.2	39
Ni _{64.1} Mo _{11.5} B _{24.4} -La(OH) ₃	323	30	55.1	19
Ni-Al ₂ O ₃ -HT	303	3.9	49.3	17
NiIr _{0.059} /Al ₂ O ₃	303	12.4	38.6	24
NiPt _{0.057} /Al ₂ O ₃	303	16.5	34	30
Ni ₃₀ Fe ₃₀ Pd ₄₀ NPs	323	21.5	40.0	33
Ni ₈₅ Ir ₁₅ @MIL-101	323	464	66.9	53
Rh ₅₈ Ni ₄₂ @MIL-101	323	344	33	44
Ni ₃ Rh ₇ /NPC-900	323	156	/	40
Pt _{0.6} Ni _{0.4} /PDA-rGO	323	2056	33.39	48

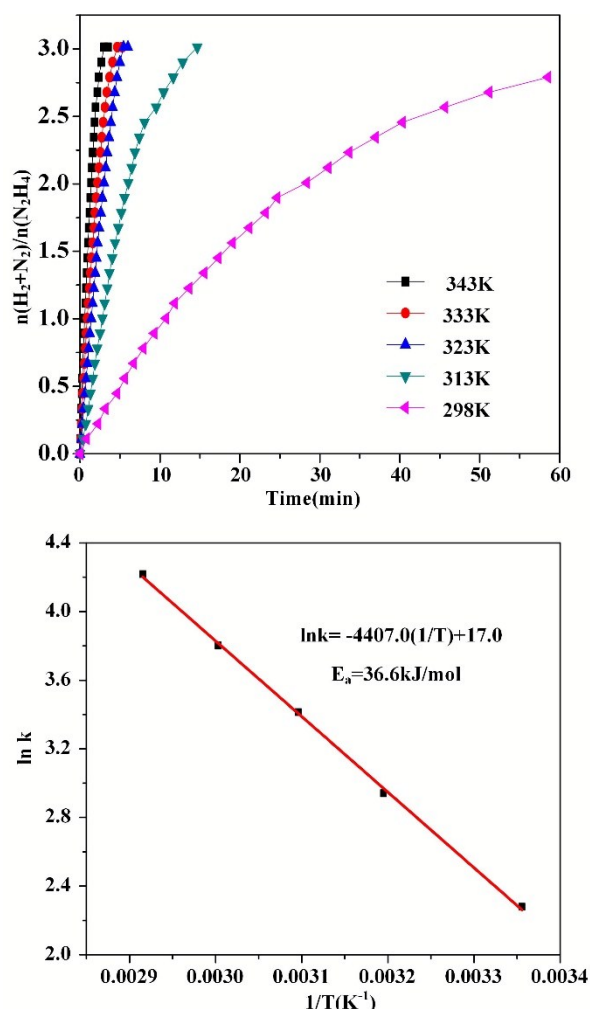


Fig. 2 (a) Time course plots for hydrogen generation by the decomposition of hydrous hydrazine by $\text{Ni}_{37}\text{Pt}_{63}/\text{g-C}_3\text{N}_4$ at 298 K, 313 K, 323 K, 333 K and 343 K. (b) Plot of $\ln k$ versus $1/T$ during the hydrous hydrazine decomposition over $\text{Ni}_{37}\text{Pt}_{63}/\text{g-C}_3\text{N}_4$ at different temperatures (catalyst=0.100 g; $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O} = 0.1$ mL).

$\text{Ni}_{82}\text{Pt}_{18}/\text{g-C}_3\text{N}_4$ are shown in Fig.S4 (ESI). A distinct peak centered at $2\theta = 27.5^\circ$ is attributed to the XRD pattern of the $\text{g-C}_3\text{N}_4$ ⁵⁷. The XRD patterns of the $\text{Ni}/\text{g-C}_3\text{N}_4$ and $\text{Pt}/\text{g-C}_3\text{N}_4$ catalysts (Fig. S4 (b) and (c), ESI) are consistent with the literature values (JCPDS, PDF 65-0380 for Ni and PDF 65-2868 for Pt), and confirm the presence of pure Ni and Pt NPs³⁶. No peaks characteristic of pure Ni and Pt were observed in the XRD patterns of $\text{NiPt}/\text{g-C}_3\text{N}_4$ with different Ni/Pt molar ratios (Fig. S4 (d-g), ESI); this reveals the existence of a bimetallic phase rather than a mixture of monometallic Ni and Pt NPs^{34-36,43,45-49}. As seen in the literature^{34-36,45-49}, the diffraction peaks of all the NiPt nanocatalysts are partially shifted compared to those of pure Ni and Pt NPs; this highlights that an alloy has formed by expanding the Ni crystal lattice through the substitution of larger Pt atoms for the smaller Ni atoms. Additionally, the morphologies of the as-synthesized $\text{Ni}_{37}\text{Pt}_{63}/\text{g-C}_3\text{N}_4$ catalysts were observed using transmission electron microscopy (TEM). The high-resolution TEM (HRTEM) image in Fig. 3(a-d) demonstrates the crystalline nature of the $\text{Ni}_{37}\text{Pt}_{63}$ NPs. The lattice spacing is calculated to be 0.210 nm, which lies between the (111) plane of face-centered cubic (fcc) Ni

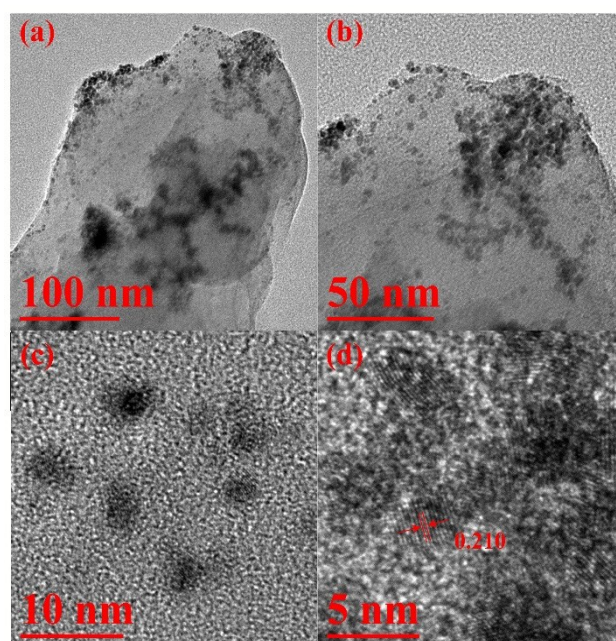


Fig. 3 (a-d) TEM images of $\text{Ni}_{37}\text{Pt}_{63}/\text{g-C}_3\text{N}_4$ with different magnification.

(0.204 nm) and fcc Pt (0.227 nm), further demonstrating the formation of an alloy structure of NiPt NPs^{34-36,43,45-49}. Fig. S5 (ESI) shows that the NiPt NPs have a narrow size distribution with a mean particle size of 3.2 ± 0.2 nm.

The elemental chemical states of the $\text{Ni}_{37}\text{Pt}_{63}/\text{g-C}_3\text{N}_4$ were elucidated using X-ray photoelectron spectroscopy (XPS). The high-resolution XPS spectra for C 1s, N 1s, Ni 2p, and Pt 4f in $\text{Ni}_{37}\text{Pt}_{63}/\text{g-C}_3\text{N}_4$ are shown in Fig. S6 (ESI). The XPS spectra of C 1s are centered at 287.8 and 284.6 eV. The first peak at 287.8 eV is attributed to the sp^2 -hybridized carbon atoms in the aromatic rings of $\text{g-C}_3\text{N}_4$, while the other peak at 284.6 eV is assigned to fortuitous carbon impurities. The N 1s spectrum of $\text{Ni}_{37}\text{Pt}_{63}/\text{g-C}_3\text{N}_4$ shows two distinct peaks at binding energies of 399.8 and 398.1 eV that are attributed to amino groups (C–N–H) and the sp^2 -hybridized nitrogen atoms of triazine (C=N–C) units, respectively. These assignments are in good agreement with the reported values for $\text{g-C}_3\text{N}_4$ ⁵⁷. Furthermore, the XPS results (Fig. S6, ESI) of $\text{Ni}_{37}\text{Pt}_{63}/\text{g-C}_3\text{N}_4$ demonstrate the presence of metallic Ni^0 with Ni 2p_{3/2} at 855.2 eV and Ni 2p_{1/2} at 872.7 eV, as well as metallic Pt^0 with Pt 4f_{7/2} 71.5 eV and Pt 4f_{5/2} 74.7 eV^{34-36,43,45-49}. The binding energies for Ni 2p and Pt 4f in $\text{Ni}_{37}\text{Pt}_{63}$ NPs were different from those of pure Ni and Pt NPs; thus, the XPS results confirm the alloy structure of $\text{Ni}_{37}\text{Pt}_{63}$ NPs in $\text{Ni}_{37}\text{Pt}_{63}/\text{g-C}_3\text{N}_4$. The formation of oxidized Ni centered at 861.1 and 879.3 eV is a result of the reported sample preparation process for XPS measurements^{43,46,49}. The results obtained from XRD, HRTEM, and XPS all demonstrate that NiPt NPs form an alloy when deposited on the surface of $\text{g-C}_3\text{N}_4$.

In conclusion, well-dispersed NiPt NPs have been successfully immobilized on $\text{g-C}_3\text{N}_4$ using a simple co-reduction method, which catalyze the dehydrogenation of hydrous hydrazine in an alkaline solution. The dehydrogenation performance of the catalysts depends on the composition of NiPt in the as-synthesized catalysts, indicating a synergistic molecular-scale alloy effect in bimetallic NiPt NPs. The $\text{Ni}_{37}\text{Pt}_{63}/\text{g-C}_3\text{N}_4$ catalysts demonstrate 100% hydrogen selectivity and

remarkable catalytic performance with a maximum TOF value of 570 h⁻¹ at 323 K, which is much higher than most of literature values for NiPt catalysts (Table 1). Designing and developing these high-performance catalysts using g-C₃N₄ as a support may accelerate the use of hydrous hydrazine as a hydrogen storage material.

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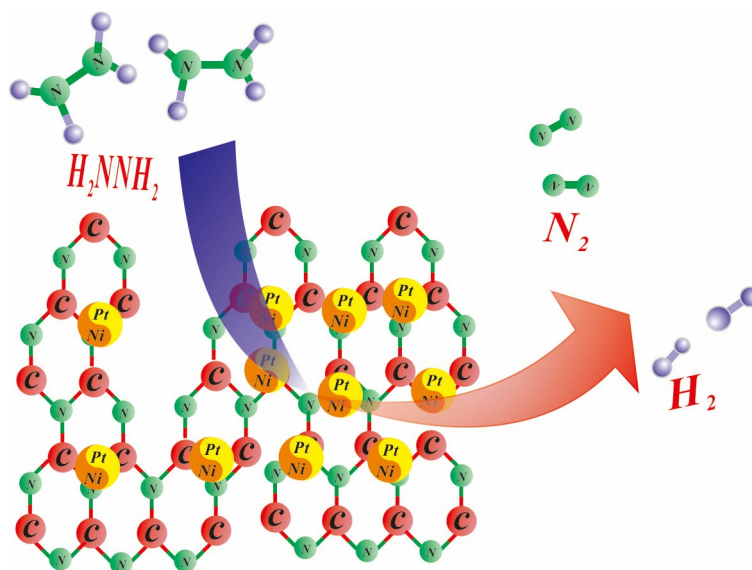
Nickel–platinum Nanoparticles Immobilized on Graphitic Carbon Nitride as Highly Efficient Catalyst for Hydrogen Release from Hydrous Hydrazine

Lixin Xu^a, Na Liu^a, Bing Hong^a, Ping Cui^a, Dang-guo Cheng^b, Fengqiu Chen^b, Yue An^b,

Chao Wan^{a,*}

^a College of Chemistry and Chemical Engineering, Anhui University of Technology, 59 Hudong Road, Ma'anshan 243002, China

^b College of Chemical and Biological Engineering, Zhejiang University, 38 Zheda Road, Hangzhou 310027, China



* Corresponding author. Tel.: +86 555 2311807; Fax: +86 555 2311822.

E-mail address: wanchao1219@hotmail.com (Chao Wan)