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To cite this article: Y Y Liu et al 2018 Nanotechnology 29 235706

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Facile preparation of surfactant-free Au NPs/
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peroxide

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Received 2 January 2018, revised 1 March 2018
Accepted for publication 23 March 2018
Published 12 April 2018

Abstract
The application of Au nanoparticles (Au NPs) often requires surface modification with chemical surfactants, which dramatically reduce the surface activity and increase the chemical contamination and cost of Au NPs. In this research, we have developed a novel Au NPs/reduced graphene oxide/Ni foam hybrid (Au NPs/RGO/NiF) by in situ reduction through ascorbic acid and replacement reaction. This method is green, facile and efficient. The Au NPs are free of chemical surfactants and are homogeneously distributed on the surface of the RGO/NiF. The as-prepared Au NPs/RGO/NiF hybrid is uniform, stable and exhibits not only a high reduction efficiency for the reduction of 4-nitrophenol with a catalytic kinetic constant of up to 0.46 min⁻¹ (0.15 cm² catalysis) but also a sensitive and selective detection of H₂O₂ with a detection limit of ∼1.60 μM.

Keywords: Au nanoparticles/reduced graphene oxide/Ni foam, catalytic properties, 4-nitrophenol, hydrogen peroxide

(Some figures may appear in colour only in the online journal)

1. Introduction
Au NPs have been widely applied in the field of catalysis [1–3] and sensing [4–6] due to their excellent optical and electrical properties. However, Au NPs prepared by a chemical method usually require surface modification with chemical surfactants to prevent their agglomeration. The presence of surfactants dramatically reduces the active sites of Au NPs, which greatly limits their performance. Moreover, during the process of application, Au NPs are difficult to use and have a low ratio of recovery. Therefore, it is necessary to find proper carrier materials to evenly disperse Au NPs in the absence of surfactants. Recently, much effort has been devoted to developing hybrid catalysts by immobilizing metal NPs onto various supporting materials, such as carbon nanotubes [7–9], graphene [10–12], Fe₃O₄ [13], TiO₂ [2, 14, 15], and polymer [16–18], which is considered an effective strategy to avoid agglomeration and improve
catalytic activity. Of particular interest is reduced graphene oxide (RGO), which is a honeycomb-like sheet of carbon with the properties of a large specific surface area, which makes it an ideal carrier of metal NPs such as Cu [19], Ag [20], Au [21, 22], Pt [23], Pd [24], for applications in catalysts and sensors. Such a kind of two-dimensional (2D) graphene sheet carrier could possibly not only improve the dispersion of Au NPs but also produce the synergistic effect to further improve their properties. Unfortunately, like other nanocatalyst hybrids of metal NPs/supported materials, metal NPs/graphene nanocomposites have the characteristic of being difficult to rapidly recycle. The highly-dispersed nanocatalysts are hard to separate totally from the system using the commonly used methods, such as magnetic adsorption and fine filtration, when there is a lot of reaction solution. Therefore, it is also important to immobilize the nanocatalyst hybrids onto a macroscopic support. Ni foam has high strength, good conductivity and a large specific surface, which makes it an excellent substrate material for nanocatalyst hybrids. Combining the advantageous properties of Au NPs/RGO and NiF is, therefore, a promising strategy to simultaneously achieve both high activity and efficient reusability by using NiF as the macro-support for Au NPs with RGO as the nano-support.

4-nitrophenol (4-NP) is a pollutant that often exists in industrial and agricultural wastewaters. It is necessary to remove it from the polluted waters to meet increasingly stringent environmental quality standards. The reduction of 4-NP to 4-aminophenol (4-AP) in the presence of a catalyst is the best method for removing the toxic 4-NP.

Diabetes has not been optimally controlled in some people. The positive monitoring and control of blood glucose are the key to curing diabetes clinically. As we know, the glucose level can be indirectly detected by testing for its reaction product, H2O2. Many studies have been carried out on the detection of H2O2. Numerous approaches have been employed for the quantitation of H2O2 including titrimation, spectrophotometry, chemiluminescence, chromatography, fluorescence, and phosphorescence [25–27]. However, most of them exhibit their own technical drawbacks such as low sensitivity and selectivity, being time-consuming, their susceptibility to interference, and needing complicated or expensive instrumentation. In order to avoid the above problems, the electrochemical technique has been widely employed in the detection of H2O2 due to its characteristics of easy operation, high sensitivity, and good selectivity. The necessary sensing materials require mass industrial production at low cost and high speed.

In this research, we developed a novel and multifunctional Au NPs/reduced graphene oxide/Ni foam hybrid (Au NPs/RGO/NiF) by in situ reduction through ascorbic acid and displacement reaction. This novel hybrid uses Ni foam as the supporting material. For application in catalysis, the sturdy structure of Ni foam ensures a long service life and a good recyclability. For application in a sensor, the continuous mesh structure of Ni foam means the as-prepared Au NPs/RGO/NiF is a free-standing monolithic electrode. In addition, Au NPs were prepared without adding any surfactant and are quickly displaced by Ni from gold salt solution. This preparation method is characterized by easy accessibility, a shorter reaction time and steady product quality. The Au NPs/RGO/NiF hybrid is used both as the catalyst for the reduction of 4-NP to 4-AP and as the nonenzymatic sensor for the detection of H2O2.

2. Experimental section

2.1. Materials and reagents

Ascorbic acid (AA) (C6H7O6, >99.70% pure), chlorauric acid (HAuCl4·3H2O, >99.99% pure), hydrogen peroxide (H2O2, 30%), 4-nitrophenol (C6H3NO3, >99.00% pure), sodium borohydride (NaBH4, >96.00% pure), glucose (C6H12O6), dopamine (DA) (C8H11O2N, >99.70%), uric acid (UA) (C4H4N3O3), citric acid (C6H8O7·H2O, >98.80% pure) and sucrose (C12H22O11, >99.99% pure), potassium ferri cyanide (K3[Fe(CN)6]), >98.00% pure), sodium hydroxide (NaOH), sodium dihydrogen phosphate (NaH2PO4, >99.50% pure), disodium hydrogen phosphate (Na2HPO4, >98.00% pure) were used in this experiment.

2.2. Preparation of Au NPs/Gr/NiF hybrid

Graphene oxide (GO) was prepared using the modified Hummer’s method. Ni foam (2 cm × 3 cm × 0.15 cm) was orderly washed with the acetone, ethanol and deionized water. Then the Ni foam was dipped into 1 M GO under the condition of ultrasound for full immersion. The Ni foam, evenly coated with GO solution, was taken out and dried at 60 °C. The above process was repeated three times, forming GO/NiF. AA (0.10 g) was dissolved in 30 ml deionized water and heated to 80 °C. GO/NiF was added to the above solution for 30 min. RGO/NiF was then obtained after washing and drying. 25 ml HAuCl4 solution (10 mM) was added to a beaker. RGO/NiF was immersed in it and taken out after only 1 min. Au NPs/Gr/NiF hybrid was prepared, washed and dried.

2.3. Characterizations and measurement

2.3.1. Reduction of 4-NP catalyzed by Au NPs/RGO/NiF hybrid

0.05 mM 4-NP solution and 0.10 M NaBH4 solution were freshly prepared. Then, 2 ml of 4-NP was mixed with 1 ml of iced NaBH4 in a bottle. The Au NPs/RGO/NiF hybrid (1 × 1 × 0.15 cm3) was immersed in the above mixed solution. At 30 s intervals, the Au NPs/RGO/NiF was removed and the remaining mix solution was transferred to a quartz cuvette for UV–vis measurement. The solution was transferred back to the bottle for sequential reaction for the next 30 s until the reaction finished.

2.3.2. Detection of H2O2 by Au NPs/RGO/NiF hybrid

For the detection of hydrogen peroxide, electrochemical measurements were performed using a CHI660D workstation with a typical three-electrode at room temperature. The Au NPs/RGO/NiF sample was used as
the working electrode, a platinum wire was used as the
counter-electrode and Ag/AgCl as the reference electrode.
Sodium phosphate buffer solution (PBS, 0.10 M, PH7.4) was
inlet with N2 for 15 min.

Raman spectra were measured by using Thermo Fisher
Raman spectrometer equipment in the range of
200−4000 cm−1. Scanning electron microscope (SEM) and
energy dispersive x-ray (EDX) spectroscopy measurements
were performed using an FEI Quanta 200 SEM with 10 KV
acceleration voltage. X-ray diffraction (XRD) spectra were
obtained using a D8-Discover x-ray diffractometer (Bruker,
Germany) by scanning at angles of 2θ=20°−80°. UV−vis
spectra were measured by a UV-2450 (Japan) spectro-
photometer with 1 cm x 1 cm x 3 cm quartz cells in the
range of 200 ~ 500 nm.

3. Results and discussion

The morphologies of RGO/NiF are examined by SEM
(figures 1(a)−(c)). Figure 1(a) shows that the RGO/NiF is a
porous structure. To verify the distribution of the elements in
the RGO/NiF, EDX element mapping was used to understand
the distribution of RGO on the NiF as shown in
figure 1(d). The result indicated that C element homo-
genously dispersed on the surface of the entire RGO/NiF.
Figure 1(e) shows the EDX spectrum of RGO/NiF hybrid and
the weight percentage and the atomic percentage of C are
calculated to be ∼4.43 and ∼18.48, respectively.

Similarly, the microstructures of Au NPs/RGO/NiF are
also detected by SEM. Figures 2(a)−(c) show the Au NPs/
RGO/NiF hybrids with different magnifications. We can see
a mass of Au NPs evenly distribute on the RGO/NiF base-
plate. The EDX-mapping shows that the C and Au elements
homogeneously dispersed over the entire Au NPs/RGO/NiF
(figure 2(d)). Figure 2(e) displays the EDX spectrum of Au
NPs/RGO/NiF hybrid and the weight percentage and the
atomic percentage of Au are calculated to be ∼4.56 and
∼1.35, respectively. The particle size distribution is shown in
figure 2(f) and the mean diameter of Au NPs is 29.76 nm.

The XRD spectra NiF, RGO/NiF, and Au NPs/RGO/
NiF hybrid are shown in figure 3. It is noted that the peaks at
2θ of 44.50°, 51.88°, and 76.43° are assigned to the (111),
(200), and (220) planes of the face-centered cubic Ni. The
RGO/NiF shows one more diffraction peak at 2θ of 26.50°,
than NiF, which is attributed to the (002) reflection of gra-
phitic carbon. In addition, the Au NPs/RGO/NiF presents
one more diffraction peak, at 38.18°, than RGO/NiF, indi-
cating the (111) planes of the face-centered cubic Au. The
XRD pattern of the Au NPs/RGO/NiF hybrid shows that Au
NPs on RGO/NiF have relatively high crystallinity.

Figure 4 exhibits the Raman spectra of NiF, RGO/NiF,
Au NPs, and Au NPs/RGO/NiF hybrid. The Raman spec-
trum of NiF has no sharp peak. The RGO/NiF presents three
peaks at ∼1350, ∼1570 and ∼2700 cm−1, indicating the
characteristic D, G and 2D bands of graphene, respectively.
The Au NPs have two outstanding peaks at ∼517 cm−1 and
∼1572 cm−1. The Au NPs/RGO/NiF hybrid presents addi-
tive peaks of all materials, indicating the successful prep-
paration of Au NPs/RGO/NiF hybrid. In addition, the stronger
Raman peaks of Au NPs/RGO/NiF are attributed to many
active Au NPs on the surface of Au NPs/RGO/NiF.

The catalytic reduction of 4-NP to 4-AP with an excess
amount of NaBH4 has often been used as a model reaction to
evaluate the catalytic performance of metal nanoparticles. A
UV−vis spectrometer is used to monitor the reaction process
in real time. Figures 5(a)−(c) show the UV−vis absorp-
tion spectra of the NiF and RGO/NiF, and Au NPs/RGO/NiF
hybrid. Obviously, the UV−vis absorption spectra of the NiF
and RGO/NiF almost have no significant changes within
60 min. Furthermore, the absorption peak at 300 nm, which is
the product of the catalyst, does not appear in the catalytic
reaction. Therefore, the NiF and RGO/NiF have almost no
catalytic effect for the catalytic reduction of 4-NP to 4-AP.
Figure 5(d) shows a linear correlation between ln(A) (A is the absorbance at 400 nm) vs reduction time, indicating that the reaction is of pseudo-first-order. Obviously, the rate constant (K) of Au NPs/RGO/NiF (0.46 min⁻¹) determined from the slope is much larger than that of NiF (3.00 × 10⁻⁴ min⁻¹) and of RGO/NiF (2.70 × 10⁻⁴ min⁻¹). We calculated the ratio (k) of the rate constant K over the macroscopic volume of the Au NPs/RGO/NiF, k = K/V. The activity k is k = 0.46 min⁻¹/(1 × 1 × 0.15 cm³) = 3.06 min⁻¹(cm³)⁻¹.
The high activity of Au NPs/RGO/NiF can be attributed to the following factors: (1) The surfactant-free Au NPs can be fully exposed at its active sites, providing a number of places for swapping elements. (2) The NiF has good conductivity and stability, speeding up electron transfer.

In the process of catalysis, other environmental factors, such as reacting temperature, the initial concentration, and the pH have an effect on the rate of catalysis. In the above testing process, the reaction was catalyzed at 25 °C in the presence of 0.05 mM 4-NP, with an unadjusted pH value of 8. In order to explore the effect of these factors on the catalytic reaction, a couple of comparisons had been carried out. The experimental results show that the temperature affects the reaction remarkably. As shown in figure 6(a), the reaction rate was significantly increased with the temperature rising and the highest rate constant is up to 32 times higher than the above result (0.46 min⁻¹). The result may be attributed to the following two reasons. (1) The high temperature reduces the reaction kinetics of the barrier. (2) The high temperature speeds up the diffusion and increases the speed of electron transfer and ion exchange in the process of the reaction. The initial concentration of 4-NP has little effect on the rate of reaction and the highest rate constant is no more than 1.5 times the 0.46 min⁻¹ (figure 6(b)). The pH has an important effect on the reaction. It is known that sodium borohydride (NaBH₄) can rapidly decompose and release hydrogen in acidic conditions, which means it is unstable in acidic conditions. However, NaBH₄ can be used in neutral and alkaline conditions. Therefore, the proper pH values are controlled within 8 to 14 and adjusted by NaOH. The result is shown in

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**Figure 5.** (a)–(c) Successive UV–vis absorption spectra of the reduction of 4-NP by NaBH₄ in the presence of NiF, RGO/NiF, and Au NPs/RGO/NiF hybrid. (d) The logarithm of the absorbance at 400 nm vs reduction time of NiF, RGO/NiF, and Au NPs/RGO/NiF hybrid.

**Figure 6.** Effect of temperature (a), initial 4-NP concentration (b), and pH (c) on the reaction rate of the reduction of 4-NP by NaBH₄ in the presence of Au NPs/RGO/NiF hybrid.
Before detecting the H$_2$O$_2$, the pH-value of the buffer solution was optimized. Figure 7 shows that pH7.4 has the highest current response. Therefore, pH7.4 was selected as the optimal pH value for the detection of H$_2$O$_2$.

In order to study the electrochemical behavior in the presence of H$_2$O$_2$, the CV evolutions of the NiF, RGO/NiF, and Au NPs/RGO/NiF were measured in N$_2$-saturated PBS (0.1 M, pH 7.4) in the potential range of −0.40 to 0.90 V at 50 mV s$^{-1}$, as is shown in figure 9(a) (in the presence of H$_2$O$_2$). Obviously, the CV of the Au NPs/RGO/NiF hybrid electrode showed a pair of redox peaks at 1.63 V and −3.60 V, respectively. On the contrary, the CVs of NiF and RGO/NiF had no obvious redox peaks (see the inset in figure 9(a)). Furthermore, the Au NPs/RGO/NiF hybrid showed a 30-fold increased range and a 100-fold increased range of current response over NiF and RGO/NiF, respectively. The reason for this is that the introduction of Au NPs increased the electrochemically active surface area and provided conductive layers for the electronic transmission. Figure 9(b) showed the CV behavior of Au NPs/RGO/NiF in the presence of H$_2$O$_2$. When added to 1 mM H$_2$O$_2$, the redox peaks of Au NPs/RGO/NiF increased with the addition of H$_2$O$_2$. Figure 9(c) presented the CV curves of the Au NPs/RGO/NiF hybrid electrode at different scan rates from 10 to 50 mV s$^{-1}$ in the presence of 3 mM H$_2$O$_2$. Clearly, along with the augmentation of scan rate, the range of current response was significantly increasing. This characteristic indicated that the redox reaction of Au NPs on RGO/NiF was rather rapid.
At the same time, the anodic current ($I_{pa}$) and cathodic peak current ($I_{pc}$) were linearly enhanced with the growing scan rate with the correlation coefficients of 0.997 and 0.989, respectively. It was indicated that the redox reaction was a quasi-reversible surface-controlled electrochemical process. The current response of the sensor was evaluated with the successive addition of H$_2$O$_2$ to a stirred electrolyte solution as a static potential of 0.1 V vs. Ag/AgCl electrode. Figure 10 shows the typical steady-state current-time ($I$-t) plot of Au NPs/RGO/NiF hybrid carried out in N$_2$-saturated PBS (pH 7.4). The top right inset is the enlarged figure of $I$-t for low concentration. The amperometric response current decreases linearly with increasing H$_2$O$_2$ concentration from 0.003 to 1.06 mM. As seen in the lower left inset, for every addition of H$_2$O$_2$, the steady state current is reached within 5 s. The linear regression equation of $I$ = −3.6891+0.1558C$_{H2O2}$/μM with a correlation coefficient of 0.998. The detection limit is 1.60 μM (at a signal-to-noise ratio of 3) and the sensitivity value is determined to be 156 μA mM$^{-1}$ cm$^{-2}$. The selectivity of the Au NPs/RGO/NiF for the detection of H$_2$O$_2$ was also evaluated. As shown in figure 11, the amperometric response of the relevant sensor upon addition of 1 mM H$_2$O$_2$, 0.5 mM glucose, 0.5 mM UA, 0.5 mM DA, 0.5 mM AA, 0.5 mM citric acid, 0.5 mM sucrose and 1 mM H$_2$O$_2$ into N$_2$-saturated PBS (0.1 M, pH 7.4). Applied potential 0.1 V.

The current response of the sensor was evaluated with the successive addition of H$_2$O$_2$ to a stirred electrolyte solution as a static potential of 0.1 V vs. Ag/AgCl electrode. Figure 10 shows the typical steady-state current-time (I-t) plot of Au NPs/RGO/NiF hybrid carried out in N$_2$-saturated PBS (pH 7.4). The top right inset is the enlarged figure of I-t for low concentration. The amperometric response current decreases linearly with increasing H$_2$O$_2$ concentration from 0.003 to 1.06 mM. As seen in the lower left inset, for every addition of H$_2$O$_2$, the steady state current is reached within 5 s. The linear regression equation of $I$ = −3.6891+0.1558C$_{H2O2}$/μM with a correlation coefficient of 0.998. The detection limit is 1.60 μM (at a signal-to-noise ratio of 3) and the sensitivity value is determined to be 156 μA mM$^{-1}$ cm$^{-2}$. The selectivity of the Au NPs/RGO/NiF for the detection of H$_2$O$_2$ was also evaluated. As shown in figure 11, the amperometric response of the relevant sensor upon addition of 1 mM H$_2$O$_2$, 0.5 mM glucose, 0.5 mM UA, 0.5 mM DA, 0.5 mM AA, 0.5 mM citric acid, 0.5 mM sucrose and a second injection of 1 mM H$_2$O$_2$. After adding the glucose, UA, DA, AA, citric acid and sucrose, the current response was
trivial. While for the addition of H$_2$O$_2$, the sensor gave a remarkable and repeatable current response. Therefore, the Au NPs/RGO/NiF hybrid-based sensor exhibits a versatile ability to not be influenced by such possible interferences coexisting along with H$_2$O$_2$.

Figure 12 illustrates the process for the preparation of Au NPs/RGO/NiF and the mechanism of the reduction of 4-NP to 4-AP and the detection of H$_2$O$_2$.

Table 1. Comparison of rate constant values for the 4-NP reduction to 4-AP and detection limit/response time for the detection of H$_2$O$_2$ by different as-prepared hybrids.

<table>
<thead>
<tr>
<th>Hybrid</th>
<th>Support material</th>
<th>Preparation time</th>
<th>Application</th>
<th>K (min$^{-1}$)</th>
<th>LOD($\mu$mol)/RT(s)</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>G0/CNT-Au</td>
<td>Graphene oxide</td>
<td>&gt;5 h</td>
<td>4-NP catalyst</td>
<td>0.042</td>
<td>—</td>
<td>[33]</td>
</tr>
<tr>
<td>G/PDA-Au</td>
<td>Graphene sheet</td>
<td>&gt;24 h</td>
<td>4-NP catalyst</td>
<td>0.225</td>
<td>—</td>
<td>[34]</td>
</tr>
<tr>
<td>GO/Au</td>
<td>Graphene oxide</td>
<td>&gt;20 h</td>
<td>4-NP catalyst</td>
<td>0.15</td>
<td>—</td>
<td>[35]</td>
</tr>
<tr>
<td>Au/graphene</td>
<td>Graphene hydrogel</td>
<td>&gt;12 h</td>
<td>4-NP catalyst</td>
<td>0.19</td>
<td>—</td>
<td>[22]</td>
</tr>
<tr>
<td>S/GO/Fe$_3$O$_4$-H$^-$</td>
<td>Graphene sheet</td>
<td>&gt;1.5 h</td>
<td>H$_2$O$_2$ sensor</td>
<td>—</td>
<td>12/~3</td>
<td>[36]</td>
</tr>
<tr>
<td>G/N/AZI/Au/GCE$^d$</td>
<td>Graphene sheet</td>
<td>&gt;5.5 h</td>
<td>H$_2$O$_2$ sensor</td>
<td>—</td>
<td>10/~3</td>
<td>[37]</td>
</tr>
<tr>
<td>Au-M-$s$G$^e$</td>
<td>Graphene sheet</td>
<td>no data</td>
<td>H$_2$O$_2$ sensor</td>
<td>2.48/~1</td>
<td>—</td>
<td>[38]</td>
</tr>
<tr>
<td>Au NPs/RGO/NiF</td>
<td>Graphene/Ni foam</td>
<td>&lt;1 h</td>
<td>Both</td>
<td>0.459</td>
<td>1.6/&lt;5</td>
<td>This work</td>
</tr>
</tbody>
</table>

* Limit of detection.  
* Response time.  
* SPCE/GS-Nafion/Fe$_3$O$_4$-Au-HRP.  
* Graphene/Nafion/AZI/Au NPs/GCE.  
* Au NPs-MWCNT-sG.

Figure 12. Schematic representation of the preparation process of Au NPs/RGO/NiF hybrid and its reduction mechanism of 4-NP to 4-AP and the detection of H$_2$O$_2$.
A linear relation is obtained between the current value and the concentration of H$_2$O$_2$ by electrochemical measurement.

In order to expound the differences between different hybrids, we made a comparison in table 1. By contrast, several advantages of the Au NPs/RGO/NiF hybrid can be found. (1) This novel hybrid uses Ni foam as the supporting material. For application in catalysis, the sturdy structure of Ni foam ensures a long service life and a good recyclability. For the sensor, the continuous mesh structure of Ni foam directly makes the as-prepared Au NPs/RGO/NiF a free-standing monolithic electrode. (2) Ascorbic acid as a green reductant meets the requirements for production technology and environmental protection. (3) Au NPs were prepared without adding any surfactant and were quickly displaced by Ni from gold salt solution. The difficult problems of agglomeration and nonuniform dispersion also have been well solved. (4) This preparation method is characterized by easy accessibility, shorter reaction time and steady product quality. It can also be applied to other noble metal nanoparticles composites, such as Pt, Ag, etc. (5) This hybrid is a new type of multifunctional material that has huge potential benefits in catalysts and biosensors.

4. Conclusions

In summary, we developed a novel Au NPs/RGO/NiF hybrid using a facile, green, and efficient method. The Au NPs are free of chemical surfactants and homogeneously distributed on the surface of RGO/NiF. The as-prepared Au NPs/RGO/NiF hybrid is uniform, stable and exhibits not only a high reduction efficiency for the reduction of 4-nitrophenol but also a sensitive and selective detection of H$_2$O$_2$. The novel Au NPs/RGO/NiF hybrid show good potential for application in both catalysts and sensors.

Acknowledgments

The authors are grateful for financial support from the Science and Technology Major Project of Shanxi Province (No. MC2016-06), National Natural Science Foundation of China [21173041], the Opening Project of Jiangsu Key Laboratory of Advanced Metallic Materials and the Fundamental Research Funds for the Central Universities and the Graduate Student Innovation Program for Jiangsu Regular Universities, China [No. KYLX16_0196]. We would also like to thank Mr. Drew Cannon from Washington State University, USA, for his help with writing in English.

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