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One-step synthesis and decoration of nickel oxide nanosheets with gold nanoparticles by reduction method for hydrazine sensing application

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Graphical abstract

Highlights:

- ✓ Synthesis of NiO nanosheets using one-step hydrothermal method and their surface modification with Au NPs.
- ✓ The surface modifications with metal endows fascinating features for sensing application.
- ✓ The hybrid (Au NPs-NiO nanosheets) nanomaterials used for the fabrication of electrochemical-based hydrazine sensors.
- ✓ An excellent sensitivity and lower detection limit were due to enhanced surface area and synergistic effect for electrocatalytic reaction.

Abstract

Morphology, nanoscale features, and tunable properties are the fundamentals of nanomaterials to many applications in use nowadays. Exciting approaches are utilized for developing and exploring new nanomaterials that enable broader impact on a variety of application areas. In this study, we synthesized thin nickel oxide (NiO) nanosheets using wet chemistry process and modified their surface with gold (Au) nanoparticles (NPs) via reduction method to obtain new hybrid (Au NPs-NiO nanosheets) nanomaterial. The morphological analysis of NiO and Au NPs-NiO nanosheets revealed small, uniform, thin, and smooth surface of NiO nanosheets formation, which become rough and decorated with small Au NPs uniformly over the NiO nanosheets surface. The synthesized NiO nanosheets and hybrid (Au NPs-NiO nanosheets) nanomaterials were further utilized to modify the glassy carbon electrode for the fabrication of electrochemical-based hydrazine sensors. The fabricated sensors were applied to detect hydrazine using cyclic voltammetry (CV). And the obtained sensing properties of the hybrid nanomaterial-based sensor were comparatively better than the only NiO nanosheets based sensors. Further, hybrid nanomaterial-based sensors characterized in detail, which showed an excellent sensitivity of $31.75 \mu\text{A nM}^{-1} \text{cm}^{-2}$. The lower detectable limit of hydrazine sensor was as low as $\sim 0.05 \text{ nM}$, which

is considerably better than the other metal oxide-based hydrazine sensors. Better sensing performance of hybrid nanomaterial-based sensor likely stems from nanosheets small size, tiny thickness, and Au modification that significantly improve the surface area and lead a positive synergistic effect (an effect arises between two or more materials that produce an enhanced effect compared to their individual effects) for electrocatalytic reaction. We believe that this hybrid nanomaterial with excellent catalytic properties could be utilized as an efficient electrode material to design other chemical and biological sensors.

Keywords: one-step synthesis; nickel oxide; nanosheets; gold nanoparticles; hydrazine sensor; excellent sensitivity

1. Introduction

Continuous progress in nanomaterials chemistry has led the development of new nanostructured nanomaterials with advanced properties for the application in different fields i.e., sensing and energy storage devices, solar cells, field-emission devices, supercapacitor, as a catalyst, and so on [1-5]. Compared with polymer and metal nanostructures, semiconducting metal oxides-based nanostructures have been used extensively, which possess controlled structural morphology [6, 7]. Therefore, many approaches have been used to synthesize nanostructures of various metal oxides (i.e., zinc oxide (ZnO), titanium oxide (TiO₂), tin oxide (SnO₂), copper oxide (CuO), nickel oxide (NiO), cobalt oxide (Co₃O₄), nickel tungstate (NiWO₄), cobalt oxide (CoO), iron oxide (Fe₂O₃), aluminum oxide (Al₂O₃), manganese dioxide (MnO₂), manganese oxide (Mn₃O₄), molybdenum trioxide (MoO₃)) using different synthesis methods [8-20]. Among all other techniques, wet chemical synthesis methods are relatively easy, cheap, and operated at low temperatures. Considering the properties of synthesized nanostructures, they are utilized in different applications.

Nanosheets like structures of metal oxide are getting attention of researcher due to their excellent surface area [10-21]. For example, Ji et al. engineered Fe_2O_3 nanosheet arrays and used for water splitting with an excellent photoelectrochemical performance [11]. Um et al. and Umar et al. utilized CuO nanosheets for photodetectors [12] and gas sensing [13] application, respectively. ZnO nanosheets were used for acetone sensing and uric acid biosensor fabrication by Li et al. [14] and Ahmad et al. [15], respectively. Park et al. and Zhu et al. used TiO_2 nanosheets for ultraviolet filtration [16] and as photoanode for enhanced photoelectrochemical performance [17], respectively. Li et al. used biodegradable MnO_2 nanosheets to reduce the cytotoxicity effect to the cells [18]. Masuda et al. used SnO_2 nanosheet assembly for cancer sensing [19]. Bai et al. synthesized rhodium oxide (Rh_2O_3) nanosheets and used as a useful electrocatalytic application [20]. Khan et al. synthesized cobalt pyrophosphate ($\text{Co}_2\text{P}_2\text{O}_7$) nanosheets and utilized them for high capacitance supercapacitors after enhancing their specific capacitance with redox additive [21].

Recently, NiO nanosheets morphology got enormous interest for the various applications due to their extremely high surface area, ultrafine thickness, remarkable properties [22-35]. For example, Wang et al. synthesized network NiO nanosheets and utilized for the construction of high-performance lithium-sulfur battery [22]. Yin et al. used NiO nanosheet network in perovskite-based solar cells as an efficient hole transporting layer [23]. Lin et al. utilized NiO nanosheets for phase conversion reaction in lithium-ion batteries, where NiO nanosheets showed electrochemical performance [24]. Zhao et al. reported use of NiO nanosheets as an efficient electrocatalyst for electrochemical water oxidation [25]. There are many reports of NiO nanosheets as an effective material for supercapacitors applications [26-31]. NiO

nanosheets were also used for gas and ion sensing [32-34]. Sun *et al.* reported synthesis of NiO nanosheets decorated on TiO₂ nanorods and used for improved photocatalytic activity [35]. Despite several applications of metal oxide nanosheets, a little research has been done on the utilization of NiO nanosheets or metal modified NiO nanosheets for sensing application. Mainly, there is no report on the use of NiO nanosheets or metal modified NiO nanosheets for hydrazine sensing.

In this research, we report for the first time, use of one-step synthesized NiO nanosheets and Au NPs modified NiO nanosheets for hydrazine detection. A wet chemistry process was used to synthesize NiO nanosheet with uniform morphology, nanoscale size, and very thin thickness. Then, the thermal reduction method was utilized to modify the surface of NiO nanosheets with Au NPs. As previously reported, the NiO nanosheets modification with TiO₂ nanorods and aluminum (Al) doping enhanced the photocatalytic and gas sensing properties, respectively [33, 35]. Since, NiO and Au NPs are themselves excellent material for hydrazine sensing. We hypothesize the NiO nanosheets modified with Au NPs will have excellent catalytic properties as well as the synergistic effect of hybrid material will enhance the sensing properties towards hydrazine. Further, Au NPs modified NiO nanosheets as hybrid material studied for hydrazine detection. The sensing properties of the sensors enhanced after Au NPs modification. The sensing properties enhancement is attributed to the small sizes of NiO nanosheets that offer high specific surface area and synergistic effect of thin nanosheets Au NPs, which were highly sensitive, stable, reproducible, and selective while detecting hydrazine. Finally, the sensing properties of the Au NPs modified NiO nanosheets are compared with the previously reported hydrazine sensors based on different nanomaterials.

2. Experimental Section

1.1. Reagents and Chemicals

Nickle(II) nitrate hexahydrate ($\geq 97\%$; $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), sodium hydroxide pellets ($\geq 98\%$; NaOH), sodium borohydride (98%; NaBH_4), gold(III) chloride trihydrate (99.9%; $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$), lysine ($\geq 98\%$), hydrazine anhydrous (N_2H_4 , 98%), uric acid (UA, $\geq 99\%$), ascorbic acid (AA, 99%), dopamine (DA), glucose (D-(+)-99.5%), potassium nitrate ($\geq 99\%$; KNO_3), potassium sulfate ($\geq 99\%$; K_2SO_4), sodium phosphate monobasic anhydrous (NaH_2PO_4 , $\geq 98\%$), sodium phosphate dibasic dihydrate ($\geq 99\%$; $\text{Na}_2\text{HPO}_4 \cdot 2\text{H}_2\text{O}$), and sodium chloride (NaCl , $\geq 99.5\%$) were used as obtained from Sigma-Aldrich. The ultra-pure water from Millipore-Q system was used to prepare phosphate buffer saline (PBS, 50 mM, pH 7.0) buffer after adding $\text{Na}_2\text{HPO}_4 \cdot 2\text{H}_2\text{O}$, NaH_2PO_4 , and NaCl (0.9%).

1.2. NiO Nanosheets Synthesis

For NiO nanosheets synthesis, two solutions of 0.6 mM $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and 1.2 mM NaOH were prepared in 60 mL deionized water, separately. Then, after mixing both solutions, it was stirred for 30 minutes at 600 rpm. In the next step, the above solution was poured into a Teflon-lined (100 mL) autoclave and heated it at 200 °C for 10h. After completion of the reaction, the obtained product was washed (several times) with deionized water using centrifugation. In the end, NiO nanosheets product was dried at 60 °C in an oven and calcined at 450 °C in air for 1h.

1.3. Modification of NiO Nanosheets with Au NPs

To enhance the surface area and generate synergistic effect from synthesized hybrid nanomaterial, we modified NiO nanosheets with Au NPs using reduction method at room temperature. In this experiment, 2.5, 5.0, and 7.5% of Au NPs were loaded on the NiO nanosheets. In the first experiment, to load 2.5% of Au NPs, 0.02 g

NiO nanosheets were dispersed in deionized water through ultrasonication for 10 minutes. Then, 0.25 mL of 0.01 M HAuCl_4 solution was added in the NiO nanosheets solution and stirred for 10 minutes. Next, 2.5 mL of 0.01 M lysine was added and further stirred for 15 minutes. Finally, 3 mL ice-cold 0.1 M NaBH_4 was added to the above solution while stirring. During this step, immediately solution color was changed, which was stirred for 30 minutes more followed by solution collection in centrifugation tubes for washing multiple times with deionized water. The obtained powder was dried in an oven at 60 °C for further use. Similarly, 5.0 and 7.5% loading were done using 0.50 and 0.75 mL of 0.01 M HAuCl_4 solution, respectively, following the above procedure.

1.4. Material Characterization

The FE-SEM (Carl Zeiss, Merlin), transmission electron microscope (TEM, FEI Tecnai G² 20 TWIN), and an X-ray diffractometer (Bruker Corporation, D8 ADVANCE, Karlsruhe, Germany) with Cu-K α radiation ($\lambda = 0.15406$ nm) in the scan range of 25-85° (scan rate, 5°/minute) were utilized to characterize the morphology and crystal structure of nanomaterials. X-ray photoelectron spectroscopy (XPS) from Kratos Analytical (AMICUS/ESCA 3400) used for elemental analysis of NiO and Au NPs-NiO nanosheets. An Al-K α X-ray source (1468.6 eV) applied at 10 kV that generated 10 mA current. Survey spectra for wide and narrow scan regions obtained at every 1 eV and 0.1 eV, respectively, while maintaining the analysis chamber pressure below 1×10^{-8} mbar.

1.5. Fabrication and Measurements of Hydrazine Sensor

To fabricate hydrazine sensor, first, GCE (3.0 mm in diameter and geometric area of 0.07065 cm²) surface was cleaned using polishing with alumina powder and sonication process. Then, 1.0 mg of synthesized nanomaterial (NiO or Au NPs-NiO

nanosheets) mixed with 1 mL butylcarbitol acetate (conducting binder) followed by sonication for 10 min. The obtained suspension (2 μ L) of NiO or Au NPs-NiO nanosheets were cast on GCE surface, dried in an oven at 60 °C for overnight, and used for hydrazine detection.

The electrochemical measurements of the hydrazine sensor electrodes were conducted using Palmsens (EmStat Blue) wirelessly connected to the computer. The electrochemical cell contains three-electrodes in it during measurement: platinum (Pt) wire as a counter electrode, the working electrode (NiO nanosheets/GCE; Au NPs-NiO nanosheets/GCE), and Ag/AgCl (saturated in KCl) as a reference electrode. The fabricated hydrazine sensors initially tested in the electrochemical probe (5 mM $[\text{Fe}(\text{CN})_6]^{3-/4-}$ containing 100 mM KCl) solution. The cyclic voltammetry (CV) measurements in the probe solution performed from 0 to 0.8 V at 50 mV/s scan rate. In the PBS buffer, CV responses were measured at different scan rate (25-250 mV/s) from -0.3 to +0.8 V. And all the sensing measurements performed at a fixed scan rate (50 mV/s).

3. Results and discussion

3.1. Materials Characterization

The morphology and composition of the as-synthesized NiO nanosheets were confirmed by FE-SEM. The FE-SEM images in Figure 1(a-c), clearly show abundant nanosheets like structures. The EDS result (Figure 1d) indicates that the nanosheets are made of Ni and O elements only. Presence of C is due to carbon tape used to fix sample and Si is from Si substrate, which was used to make a thin film for characterization. No other peak of impurity was noticed. Further, XRD and TEM were utilized to confirm the degree of crystallinity and to present a clear morphology

of nanosheet, as shown in Figure 2. The XRD pattern (Figure 2a) exhibits five diffraction peaks at 37.4° , 43.2° , 63.1° , 75.5° , and 79.4° corresponding to the (111), (200), (220), (311), and (222) crystal planes, respectively, of cubic NiO phase (JCPDS No. 47-1049). Figure 2b and 2c shows the TEM images of NiO nanosheets. It can be seen that nanosheets uniformly grown in shape with small size, such as length (~ 135 nm), diameter (~ 115 nm), and small thickness. The HRTEM image (Figure 2d) and selected-area electron diffraction (SAED, see inset of Figure 2d) pattern, further confirm the crystalline nature of nanosheets, which is also consistent with the XRD result.

The FE-SEM and TEM measurements carried out then to investigate the Au NPs modification of NiO nanosheets (Figure 3). As shown in high-resolution FE-SEM images in (Figure 3), the Au NPs anchored on the surface of NiO nanosheets, but that were not uniform for 2.5% Au NPs modified. The 5.0% Au NPs modified NiO nanosheets surface was uniformly decorated with NPs. However, 7.5% Au NPs modified NiO nanosheets surface coated with smaller and bigger NPs. The formation of bigger NPs is ascribed to the large percentage of Au precursor in the reaction solution, which formed cluster like NPs on the NiO nanosheets surface. The Au NPs modified NiO nanosheets surface was further evidenced by TEM observation (Figure 3c, 3f, and 3i), which were consistent with the FE-SEM observations.

The elemental analysis of NiO nanosheets and compositional change after Au NPs decoration on NiO nanosheets are further characterized by XPS (Figure 4). As shown in Figure, the survey spectra of NiO nanosheets presented peaks of Ni 2p and O 1s. For Au NPs modified NiO nanosheets, one additional peak of Au 4f was present, which is ascribed to the Au NP [36, 37]. Figure 4b shows the high-resolution spectra of Au 4f for Au NPs modified NiO nanosheets sample, where two peaks of

Au 4f_{7/2} and Au 4f_{5/2} lines are located at around 83.5 eV and 87.5 eV. The deconvolution lines of high-resolution Ni 2p spectrum in NiO nanosheets (Figure 4c) and Au NPs modified NiO nanosheets (Figure 4d) are shown. In NiO nanosheets sample, peaks correspond to the presence of two Ni 2p_{3/2} and two 2p_{1/2} lines. They are located at approximately 862, 866, 869.5 and 882 eV in Ni 2p high-resolution spectra. However, in Au NPs modified NiO nanosheets sample (Figure 4d) peaks were present at approximately 853, 856, 860 872,5 and 878.5 eV that represents deconvolution lines of Ni 2p_{3/2} and 2p_{1/2} high resolution spectra. This confirms the coexistence of both NiO and Au NPs. Figure 4e and 4f shows the high-resolution of O 1s spectra comprises two peaks for NiO nanosheets and Au NPs modified NiO nanosheets samples, respectively. In NiO nanosheets sample, the deconvolution of O 1s spectra shows that the peaks are located at 538 and 541 eV corresponding to Ni-O bond and C-O bond, respectively. Similarly, the peak of Ni-O bond was located at 528 eV while C-O bond was detected at 531 eV in O 1s spectra of Au NPs modified NiO nanosheets sample. The bonding energies of peaks in high resolution spectra of Ni 2p and O 1s exhibits that peaks shift to the lower level of spectra after the localization of Au NPs on the surface of NiO nanosheets [38, 39]. Ni 2p_{3/2} and Ni 2p_{1/2} peaks in Ni 2p spectra and Ni-O, C-O peaks in O 1s spectra also follow this trend. Collectively, FESEM, TEM, and XPS studies confirm the successful modification of NiO nanosheets with Au NPs.

3.2. Evaluation of electron-transfer characteristics

To evaluate the electron-transfer characteristic of modified electrodes, CV response of the bare GCE (i), bare NiO nanosheets/GCE (ii), 2.5% Au NPs-NiO nanosheets/GCE (iii), 5.0% Au NPs-NiO nanosheets/GCE (iv), and 7.5% Au NPs-

NiO nanosheets/GCE (v) in 5.0×10^{-3} M $[\text{Fe}(\text{CN})_6]^{3-/4-}$ solution having 100×10^{-3} M KCl (scan rate, 50 mV/s), as shown in Figure 5. All CV response are measured in a constant potential range (0 V to 0.8 V) and at a fixed scan rate of 50 mV/s. Compared to the bare GCE, an enhanced current response is obtained for all nanomaterial modified electrodes. The current response of the Au NPs-NiO nanosheets modified electrode was better, which is due to Au NPs modification on the nanosheets surface where metal NPs have the special active sites that adsorb more electrons and transfer to the nanosheets surface. Importantly, 5.0% Au NPs-NiO nanosheets modified GCE showed the best current response. However, a higher amount of Au NPs (7.5%) fixed on NiO nanosheets modified electrode showed decreased current response, which may be due to the clustering/aggregating of Au NPs on nanosheets surface that reduce the surface area, bury their active sites due to cluster formation, and reduce the electron transfer rate. Hence, we selected 5.0% Au NPs-NiO nanosheets modified GCE for further sensing characterizations.

3.2. Electrocatalytic properties of modified electrodes

To check the effectiveness of the NiO nanosheets and Au NPs modified NiO nanosheets, the electrochemical behavior of the electrodes evaluated in the absence and presence of hydrazine (Figure 6). As shown in Figure 6a, in the absence of hydrazine there was no apparent peak in the CV responses for all the electrodes. However, in the presence of 25 nM hydrazine, the bare GCE showed very little response compared with NiO nanosheets and Au NPs-NiO nanosheets modified electrodes (curve ii-v, Figure 6b). The increased current response is attributed to the hydrazine oxidation over the nanomaterial surface that facilitates electron transfer. During the electro oxidation of hydrazine over Au NPs-NiO nanosheets modified

electrode, OH^- adsorbed onto the Ni of NiO and electrons of OH^- were localized to 3d orbital of Ni, where hydrazine reacts with adsorbed OH^- and produces N_2 and H_2O . Additionally, the presence of Au NPs over NiO nanosheets offers an excellent catalytic surface for hydrazine oxidation. As expected, 5.0% Au NPs-NiO nanosheets modified GCE showed the maximum current response, which was latter used for sensing response measurements. In the 7.5% Au NPs-NiO nanosheets modified electrode, the current response was lower due to cluster formation that offers less active sites and reduced catalytic activity during hydrazine sensing.

For further electrode and solution interface behavior characterization, the CV response of the sensor (5.0% Au NPs-NiO nanosheets/GCE) was measured at different scan rate (i.e., 25-250 mV/s) in the presence of 25 nM hydrazine in PBS (50 mM, pH 7.0) and the results are depicted in Figure 7a. From the CV response, it can be observed that the hydrazine oxidation peak current was increased with the increasing the scan rate. Figure 7b show the calibrated plot of peak current vs. scan rate. The obtained plot was linear with linear regression coefficient (R^2) of 0.994, which further indicate the surface-confined electrocatalytic oxidation process of hydrazine.

For further sensing parameters characterization, CV response with increasing hydrazine concentration (0.1-150 nM) was measured (Figure 8a). An overall linear current response is shown in Figure 8b. The calibrated curve showed linearity in the range of 0.1-110 nM and high sensitivity ($31.75 \mu\text{A nM}^{-1}\text{cm}^{-2}$) with linear regression coefficient (R^2) value of 0.9992 (Figure 8b). An ultra-low detection limit of ~ 0.05 nM was obtained, which is better than many reports (see Table 1) based on metal and metal oxides [40-54]. The high sensitivity and low limit of detection can be ascribed to the excellent surface area of NiO nanosheets and Au NPs modification that

provided positive synergistic effect during the electrocatalytic reaction. The ultra-high sensitivity and detection limit is also attractive for the utilization of the sensor for the quantitative measurement of hydrazine in water samples with low hydrazine concentrations.

3.3 Selectivity, reproducibility, and stability tests

The selectivity test of fabricated hydrazine sensor was measured using CV in PBS containing 25 nM hydrazine in the absence (curve i), presence of low (25 nM, curve ii), and high (50 nM, curve iii) concentration of each interfering species such as AA, glucose, DA, UA, KNO₃, K₂SO₄ (Figure 9a). From the CV response, it can be seen a similar current response for 25 nM hydrazine in the absence and presence of interfering species, further confirming selectivity nature of fabricated hydrazine sensor. Five similar fabricated electrodes showed almost similar current response during 25 nM hydrazine detection, stating high reproducibility of electrodes (Figure 9b). Further, the cyclic stability of the hydrazine sensor electrode was measured in 25 nM hydrazine (Figure 9c). After 50 cycles, there was a slight decrease (~4.8%) in the current response. Overall, it can be concluded that fabricated sensors are selective, reproducible, and stable for electrochemical detection of hydrazine.

4. Conclusions

In conclusion, the present study describes the one-step synthesis of NiO nanosheets and successful surface modification of NiO nanosheets with Au NPs. This surface modification step is vital for an application (i.e., hydrazine sensing), where Au NPs enhance the surface area and provide synergistic effect during the electrocatalytic reaction. Initially, the electron transfer study of the fabricated

hydrazine sensors was conducted to know an efficient electrode, where enhanced electron transfer takes place between GCE and fixed nanomaterials. We found out that 5.0% Au NPs-NiO nanosheets attached onto GCE showed better electron transfer as compared to the other electrodes. Further, 5.0% Au NPs-NiO nanosheets/GCE sensors were selected as an efficient sensor and utilized to detect hydrazine. The fabricated electrode showed an excellent sensitivity of $31.75 \mu\text{A nM}^{-1}\text{cm}^{-2}$ in the linear range of 0.1-110 nM and ultra-low detection limit of ~ 0.05 nM. Better sensing performance, good reproducibility, cyclic stability, and selectivity confirms the reliable and promising application for hydrazine detection. The excellent electrocatalytic reaction for hydrazine demonstrates that these Au NPs modified NiO nanosheets can also be applied for other sensing applications.

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Table 1. Hydrazine sensing performance of different nanomaterials.

Material/electrode	Detection technique	Sensitivity ($\mu\text{A}\mu\text{M}^{-1}\text{cm}^{-2}$)	Linear range (μM)	Detection limit (nM)	Ref.
NiO nanostructures/GCE	Amperometry	-	0.1-0.5	5	[40]
NiO nanostructures/GCE	Amperometry	-	0.6-1.6	5	[40]
Nafion/ZnO/Au	Amperometry	8.56	0.1-1.2	200	[41]
ZnO nanorods/Ag	FET	59.175	0.001-60	~3.86	[42]
CoOOH nanosheets/cobalt	Amperometry	0.155	up to 1200	20,000	[43]
Cu NPs-PANI-GO	Amperometry	0.35993	0.04-0.48	4.5	[44]
Au NP-MWCNT/GCE	Amperometry	4.98	0.1-100	30	[45]
rGO-Co ₃ O ₄ @Au	Amperometry	0.58304	10-620	443	[46]
Au-Pd NCRs/GCE	Amperometry	128.728	315-501	20	[47]
Au-Pd NCRs/GCE	Amperometry	67.906	0.1-295	20	[47]
Au@Pd/CB-DHP/GCE	Amperometry	0.0824	2.5-88	1770	[48]
Pd/rGO/RDE	Amperometry	-	0.04-200	7	[49]
GO/CTS/Pt	Amperometry	0.1046	0.02-10000	3600	[50]
TiO ₂ NTs-Au@Pd/GCE	Amperometry	0.013	0.06-700	12	[51]
ZnO nanorods/Ag	CV	105.5	0.01-98.6	5	[52]

Co/MOF/MPC/GCE	Amperometry	1750	0.5-15	210	[53]
Co/MOF/MPC/GCE	Amperometry	1060	15-235	210	[53]
Au nanocage/CMG/GCE	Amperometry	0.038	6-30	500	[54]
Au nanocage/CMG/GCE	Amperometry	0.01	30-1700	500	[54]
Au NPs-NiO nanosheets/GCE	CV	31750	0.0001-0.110	~0.05	This work

Abbreviations: PANI-polyaniline; MWCNT-multiwalled carbon nanotubes; NCRs-nanorod chains; CB-carbon black; DHP-dihexadecylphosphate; Pd-palladium; rGO-reduced graphene oxide; RDEs-rotating disk electrodes; CTS-chitosan; GO-graphene oxide; NTs-nanotubes; Co-cobalt; MOF-metal-organic framework; MPC- macroporous carbon; chemically modified graphene (CMG).

Figure captions:

Figure 1. (a) Low-, (b) high-resolution FE-SEM images of NiO nanosheets, FE-SEM image of NiO nanosheets (c) and corresponding EDS result (d).

Figure 2. (a) XRD pattern, (b-c) low-resolution TEM, and (d) high resolution TEM images of NiO nanosheet. Inset b and d show the scheme of nanosheet and SAED pattern, respectively.

Figure 3. Analysis of Au NPs modification of NiO nanosheets. FE-SEM images of (a-b) 2.5%, (d-e) 5.0%, and (g-h) 7.5% Au NPs modified NiO nanosheets. TEM images of (c) 2.5%, (f) 5.0%, and (i) 7.5% Au NPs modified NiO nanosheets. Schemes of modified sheets are shown in insets of c, f, and i.

Figure 4. XPS survey spectra of NiO and Au NPs modified NiO nanosheets (a), high resolution spectra of Au 4f obtained from Au modified NiO nanosheets (b), high resolution spectra of Ni 2p obtained from (c) NiO nanosheets and Au modified NiO nanosheets (d), high resolution spectra of O 1s obtained from (e) NiO nanosheets and Au modified NiO nanosheets (f).

Figure 5. (a) CV response of the bare GCE (i), bare NiO nanosheets/GCE (ii), 2.5% Au NPs-NiO nanosheets/GCE (iii), 5.0% Au NPs-NiO nanosheets/GCE (iv), and 7.5% Au NPs-NiO nanosheets/GCE (v) in 5.0×10^{-3} M $[\text{Fe}(\text{CN})_6]^{3-/4-}$ solution having 100×10^{-3} M KCl (scan rate, 50 mV/s). (b) Calibrated histogram of CV responses i.e. peak current vs. electrodes.

Figure 6. CV response of the bare GCE (i), bare NiO nanosheets/GCE (ii), 2.5% Au NPs-NiO nanosheets/GCE (iii), 5.0% Au NPs-NiO nanosheets/GCE (iv), and 7.5% Au NPs-NiO nanosheets/GCE (v) in the absence (a) and presence (b) of 25 nM hydrazine in PBS (50 mM, pH 7.0).

Figure 7. CV response of 5.0% Au NPs-NiO nanosheets/GCE in the presence of 25 nM hydrazine in PBS (50 mM, pH 7.0) at different scan rate i.e. 25 to 250 mV/s (a) and calibrated response i.e. peak current vs. scan rate (b).

Figure 8. (a) CV response of 5.0% Au NPs-NiO nanosheets/GCE with increasing hydrazine concentration from 0.1 nM to 150 nM in PBS (50 mM, pH 7.0) at 50 mV/s and (b) corresponding linear calibration curve i.e. current response vs. hydrazine concentration.

Figure 9. (a) Selectivity test of the hydrazine sensor (5.0% Au NPs-NiO nanosheets/GCE). CV response in (i) only 25 nM hydrazine, (ii) 25 nM hydrazine and 25 nM of each interfering species, and (iii) 25 nM hydrazine and 50 nM of each interfering species in PBS (50 mM, pH 7.0) at 50 mV/s; (b) Reproducibility test. CV response of five similar fabricated electrodes in 25 nM hydrazine in PBS (50 mM, pH 7.0) at 50 mV/s; and (c) Cyclic stability test. CV response of the sensor in 25 nM hydrazine at 50 mV/s.

Figures:

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