Research Article
A Sensitive Resonance Rayleigh Scattering Method for Na⁺ Based on Graphene Oxide Nanoribbon Catalysis

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The gold nanoparticle reaction of HAuCl₄·H₂O₂ was very slow under 60° C, and the as-prepared graphene oxide nanoribbons (GONRs) exhibited strong catalysis of the reaction to form gold nanoparticles (AuNP) that appeared a resonance Rayleigh scattering (RRS) peak at 550 nm. Upon addition of potassium pyroantimonate (PA) ligand, it was adsorbed on the GONRs surface to inhibit the catalysis to cause the RRS peak decreasing. When the analyte of Na⁺ was added, the coordination reaction between PA and Na⁺ took place to form the stable complexes of [Na₂(PA)] to release free GONRs catalyst that resulted in the RRS peak increasing linearly. Accordingly, a new and sensitive RRS method for Na⁺ was established, with a linear range of 0.69-25.8 nmol/L and a detection limit of 0.35 nmol/L Na⁺.

1. Introduction

Graphene oxide nanoribbons (GONRs), prepared by the oxidative dissociating of multiwalled carbon nanotubes (MWCNTs), show novel physical, chemical, and catalytic properties and good solubility [1–5] and have been utilized in nanoanalysis. Sun et al. [6] synthesized GONRs based on the decompression of MWCNTs by microwave energy, and a core shell MWCNT/GONR modified glass carbon electrode was fabricated to detect 0.1-8.5 μmol/L ascorbic acid, 0.15-12.15 μmol/L dopamine, and 0.15-11.4 μmol/L uric acid simultaneously, with detection limits of 0.06 μmol/L, 0.08μmol/L and 0.07μmol/L respectively. Zhu et al. [7] prepared a core–shell heterostructure MWCNTs@GONRs by partially unzipping of MWCNTs from longitudinal side with a simple wet chemical strategy and applied it for electrochemical determination of three kinds of polycyclic aromatic amine (PAAs). Zhang et al. [8] carried out PtPd-rGONRs nanocubes by fixing GONRs on the PtPd concave through a hydrothermal process. The PtPd-rGONRs-based electrochemical detection platform can be applied to 0.01-3μg/mL trinitrotoluene (TNT) detection in tap water and lake water samples, with a detection limit of 0.8 ng/mL. Up to date, there is rare report about using GONRs to detect sodium by resonance Rayleigh scattering (RRS) method.

RRS is simple, rapid, and sensitive and has been used in a wide range of applications in different fields such as biochemistry, analytical chemistry, and nanomaterial research [9–14]. Liang et al. [15] reported a RRS-ET method for the analysis of fluorine. Fluorine ions react with fluorine reagent (FR) and La(III) to generate blue ternary complex that exhibited strong absorption at about 370 nm. Upon addition of graphene oxide/nanogold (GO/NG) as RRS spectral probe with strong RRS peak at 370 nm, and the RRS intensity decreased with the increase of fluorine ion concentration due to the RRS energy transfer (RRS-ET). Its linear range was 6.0×10⁻⁸-1.3×10⁻⁵ mol/L, with a detection limit of 3.0×10⁻⁸ mol/L. Wang et al. [16] prepared graphene oxide/gold nanoparticle (GO/GN) composites by citrate reduction that exhibited a strong resonance RS (RRS) peak at 370 nm, and 0.025-5 mM KIO₃ and 0.5-100 mM H₂O₂ can be determined, respectively. Yang et al. [17] reported a RRS method for simultaneous determination of D- L-tryptophan chiral enantiomer complexed with β-Cyclodextrin. Sodium ions play an important
role in life activities. Most of it exists in the extracellular and bone; it plays a role in regulating the body’s osmotic pressure, acid-base balance, muscle, and heart activity. The imbalance of sodium ions in the body can cause some discomfort, including convulsions, hypotension, vomiting, heart failure, and kidney failure. Therefore, the detection of Na\(^+\) has become an important issue. The common methods for the determination of Na\(^+\) are potentiometric titration [18], flame atomic emission spectrometry [19], temperature titration [20], atomic absorption spectrometry [21], ion chromatography [22], and inductively coupled plasma mass spectrometry [23]. Some of these methods have the advantages of low sensitivity, complicated operation, large ion interference, and many affecting factors. In this paper, the as-prepared GONRs were used as catalyst, and a rapid and sensitive RRS method was established for detecting Na\(^+\), based on the ligand regulating GONRs catalytic activity.

2. Results and Discussion

2.1. Analytical Principle. Under the selected conditions, GONRs had strong catalysis on the reduction of HAuCl\(_4\) by H\(_2\)O\(_2\) which was very slow without a catalyst. With the increase of GONRs concentration, the reaction was obviously enhanced. The concentration of formed gold nanoparticles in the system was increased, which showed a strong RRS peak. Potassium pyroantimonate (PA) binding on GONRs surface had an inhibitory effect on the catalysis of GONRs. Moreover, Na\(^+\) could react with CAP to form stable complexes Na\(^+\)CAP, thus releasing the binding of CAP to GONRs and restoring the catalysis of GONRs to cause the RRS peak increasing. Accordingly, a new RRS method for Na\(^+\) by ligand regulating GONRs catalytic activity was established (Figure 1).

2.2. RRS Spectra. The CH\(_3\)CH\(_2\)OH-Na\(^+\)-PA-GONR-H\(_2\)O\(_2\)-HAuCl\(_4\) system exhibited three RRS peaks at 305 nm, 370 nm, and 550 nm that are ascribing to surface plasmon resonance effect of AuNPs (Figure 2). With the concentration of Na\(^+\) increased, the RRS peaks increased linearly. Although both peaks at 305 nm and 370 nm are more sensitive than the peak at 550 nm, the latter is free from interference of benzene compounds and was selected for determination of sodium ions. While the CH\(_3\)CH\(_2\)OH-Na\(^+\)-PA system has two weak peaks at 295 nm and 370 nm, and the RRS signals increased with Na\(^+\) concentration increase slowly (Fig. S1). Therefore, the contribution of [Na\(_2\)(PA)] on the system can be excluded. The CH\(_3\)CH\(_2\)OH-Na\(^+\)-PA-GO-H\(_2\)O\(_2\)-HAuCl\(_4\)-VB4r system generated the characteristic nanogold peaks at 370 nm and 545 nm (Fig. S2). The RRS spectra of different analysis systems were studied, and the intensity of RRS spectra increased gradually with the increase of the amount of analyte. The binding of GONR and PA inhibit the catalysis when there was no Na\(^+\) (Fig. S3, S4). In a certain range, with the increase of GO, GONR concentration, the reaction of H\(_2\)O\(_2\)-HAuCl\(_4\) system was enhanced, so the concentration of nanoparticles generated by the reaction increased and the RRS signals of the system gradually increased (Fig. S5, S6).

2.3. Absorption Spectra. According to the procedure, the UV-vis absorption spectra of analytical systems were recorded. There was an absorption peak at 580 nm of the CH\(_3\)CH\(_2\)OH-Na\(^+\)-PA–GONR/GO-H\(_2\)O\(_2\)-HAuCl\(_4\) system, and the peak gradually increased and narrowed with the increase of Na\(^+\)
concentration in a certain range (Figure 3, Fig.S7). The binding of GONR and PA inhibits the catalysis when there was no Na⁺ (Fig. S8, Fig.S9). As the concentration of GONR/GO increased, the catalytic activity increased and more gold nanoparticles were formed, and the absorption of the system gradually increased (Fig. S10, Fig. S11).

2.4. Catalysis and Inhibition. According to the experimental conditions, GO/GONR can catalyze the reaction of H₂O₂-HAuCl₄ to form gold nanoparticles. In a certain range of concentration, the catalytic ability was enhanced with the catalyst increase and the formed gold nanoparticles were increased. The RRS intensity of 550 nm increases linearly and the corresponding UV absorbance has a certain degree of enhancement to catalyst concentration. At the same time, the catalytic effects of different quantum dots were compared. (Table 1). GONR-H₂O₂-HAuCl₄ has the strongest catalytic ability. The PA attaches to the surface of the catalyst, preventing contact of the catalyst with the reactants when PA is added to the system. As the amount of PA increased, the catalytic RRS/UV effect was attenuated, in which the system ΔI₅₀nm was negatively correlated with PA concentration (Table 1).

2.5. Scanning Electron Microscopy (SEM). According to the selected conditions, 0.1mL reaction solution was taken and diluted 100 times in a 10 mL centrifuge tube. Then 10 μL solution was dropped onto the silicon wafer and naturally dried, and SEM of the sample was obtained. For the Na⁺-PA-GONR-H₂O₂-HAuCl₄ system, the binding of GONR and PA inhibits the catalysis when there was no Na⁺. So the reaction of H₂O₂ reducing HAuCl₄ at 60°C was slower, and the produced nanogold in the solution was less (Figure 4(a)), with an average diameter of 150 nm. When Na⁺ was added, GONR was not completely bound and had some catalysis, resulting in a certain amount of nanogold with irregularly shaped particles with the average diameter of about 100 nm (Figure 4(b)).

2.6. Optimization of Analytical Conditions. The analytical conditions were optimized, respectively (Fig. S12). The effect of GONRs concentration on the system was investigated and the ΔI values increased rapidly at first and reached the maximum when the concentration of GONR was 15.8 μg/mL. So, 15.8 μg/mL GONR was chosen. The ΔI values of SERS increased rapidly at first and decreased after the maximum with CAP concentration increased. According to the result, 3.14 μmol/L CAP was chosen. H₂O₂ was a reducer for the redox reaction and when the concentration of H₂O₂ was 3.3 mmol/L, ΔI was the largest. So 3.3 mmol/L H₂O₂ was chosen. The effect of HAuCl₄ concentration was investigated according to the procedure and when HAuCl₄ concentration was 0.193 mmol/L, ΔI was the largest. So 0.193 mmol/L HAuCl₄ was selected. The effect of reaction temperature was investigated in the range of 20-80°C water bath. ΔI enhanced sharply with the temperature increasing at 40-60°C and tended to be stable before and after that. So, a water bath of 60°C was used. The effect of reaction time was investigated. ΔI got the maximum and tended to be stable when the reaction time was 20 min. So, 20 min was chosen.

2.7. Working Curve. The working curves between Na⁺ concentration and their corresponding ΔI were plotted for the GONR and GO two analytical systems. Results show that the GONR system was more sensitive than the GO, with a linear range of 0.69-25.8 μmol/L, regression equation of ΔI = 85.4C_{Na⁺}+244.1, a coefficient of 0.9712, and detection limit of 0.35 nmol/L Na⁺ and was selected to determine Na⁺ in sample.

2.8. Influence of Interfering Ions. The interference of common coexistence on the determination of 1.72 nmol/L Na⁺ was investigated within a relative error of ±10%. Results showed that 100 times Ca²⁺, Ni²⁺, Bi³⁺, NH₄⁺, SO₄²⁻, Pb²⁺, Zn²⁺, NO₂⁻, glutamic acid, aspartic acid, valine, phenylalanine, 80 times Cr⁶⁺, Mg²⁺, Al³⁺, K⁺, 50 times Ba²⁺, Fe³⁺, Mn²⁺, and 10 times Cu²⁺ had no interference on the determination of Na⁺. Therefore, the method had good selectivity.

2.9. Sample Analysis. The drinking water samples were purchased from supermarket and filtered by filter paper. According to the procedure, Na⁺ content was detected in the samples. The RRS results were in agreement with that of atomic absorption spectrometry (AAS), the recovery was 96.5% to 98.2%, and relative standard deviation is 3.3% to 6.7% (Table 2).

3. Materials and Methods

3.1. Apparatus. A model of Hitachi F-7000 fluorescence spectrophotometer (Hitachi High-Technologies Corporation, Japan) by synchronous scanning technique with a volt of 400 v, excited slit and emission slit of 5 nm, emission filter of 1% T attenuator and λₐₓ-λₑₓ=Δλ=0, a model of TU-1901 double beam UV-visible spectrophotometer (Beijing
Table 1: Comparing of the catalytic and inhibition characteristics by RRS method.

<table>
<thead>
<tr>
<th>System</th>
<th>Regress equation</th>
<th>Linear range</th>
<th>Coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td>GONR</td>
<td>$\Delta I_{550nm} = 50.8 \times C_{GONR} + 26.7$</td>
<td>$0.9868$</td>
<td></td>
</tr>
<tr>
<td>GO</td>
<td>$\Delta I_{445nm} = 34.2 \times C_{GO} + 130.2$</td>
<td>$0.9768$</td>
<td></td>
</tr>
<tr>
<td>PA-GONR</td>
<td>$\Delta I_{550nm} = 241.8 \times C_{PA} - 8.6$</td>
<td>$0.983$</td>
<td></td>
</tr>
<tr>
<td>PA-GO</td>
<td>$\Delta I_{545nm} = 150.4 \times C_{PA} + 130$</td>
<td>$0.9537$</td>
<td></td>
</tr>
</tbody>
</table>

Table 2: Analytical results of Na$^+$ in samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Detection (ng/L)</th>
<th>Average (ng/L)</th>
<th>Added (ng/L)</th>
<th>Found (ng/L)</th>
<th>Recovery (%)</th>
<th>RSD (%)</th>
<th>Content (mg/L)</th>
<th>AAS (mg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample1</td>
<td>205.9, 198.6,</td>
<td>204.9</td>
<td>367.6</td>
<td>558.6</td>
<td>97.6</td>
<td>6.7</td>
<td>2.1</td>
<td>2.0</td>
</tr>
<tr>
<td></td>
<td>185.9, 211.3,</td>
<td>222.6</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>156.9, 157.3</td>
<td>144.2, 152.1,</td>
<td>148.6</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample2</td>
<td>151.8</td>
<td>367.6</td>
<td>510.2</td>
<td>98.2</td>
<td>3.6</td>
<td>1.5</td>
<td>1.6</td>
<td></td>
</tr>
<tr>
<td>Sample3</td>
<td>101.3, 99.7, 102.5, 106.9, 98.1</td>
<td>101.7</td>
<td>367.6</td>
<td>453.1</td>
<td>96.5</td>
<td>3.3</td>
<td>1.8</td>
<td>1.9</td>
</tr>
</tbody>
</table>

3.2. Reagents. A 11.8 mmol/L potassium pyroantimonate (PA) solution: 1.5 g of potassium pyroantimonate (AR, Shanghai Reagent Four Hewei Chemical Co., Ltd.) was added to a conical flask before 150 mL distilled water and 0.15 g KOH was added and boiled to dissolve. After cooling to room temperature, the solution was transferred into a 250 mL volumetric flask and added water to the mark. It was diluted 100 times for use. A 2.9 mmol/L HAuCl$_4$ (National Pharmaceutical Group Chemical Reagents Company, China), 10 μmol/L VB4r, 10 mol/L H$_2$O$_2$, KMnO$_4$ (s), multiwall carbon nanotube (MWCNTs, short, 8-15 nm, 95% purity, length of 0.5-2 μm, 8-15 nm in diameter, No. XFM10, XFNANO, Nanjing), 50% CH$_3$CH$_2$OH, 50 mmol/L NaCl standard solution, 39.33 μmol/L sodium pyroantimonate solution (118 μmol/L potassium pyroantimonate solution and 129 mmol/L NaCl solution were mixed in a volume ratio of 1:2 according to the desired volume) were prepared. A 1.0 mg graphene oxide (GO) was dissolved in 100 mL water with ultrasonic treatment to obtain 10 μg/mL GO solution. It was treating for 15 min by ultrasonic process before use. All reagents were of analytical grade and the water was doubly distilled.

GONRs were prepared by the dissociating of MWCNTs. 50 mg MWCNT powder was added to a 50 mL round bottom flask containing 10 mL of concentrated H$_2$SO$_4$ before reacting...
for 1 h. Then 300 mg of KMnO₄ was added to the solution, mixed well, and heated at 60 °C for 2 h. Next, the product was poured into 200 mL of ice water containing 5 mL of 30% H₂O₂. Then the solution was ultrasonicated for 10 min before centrifuging at 7000 rpm for 10 min. The supernatant was taken and the manganese dioxide generated in the reaction was removed. The final concentration of GONRs solution was 238 μg/mL. It was neutralized with 50 mmol/L NaOH and then diluted to the desired concentration before use.

3.3. Procedure. A 100 μL 50% CH₃CH₂OH, 50 μL L118 μmol/L PA and a certain amount of NaCl standard solutions were added to a 5 mL marked test tube, mixed well, and reacted for 10 min at room temperature. Then a 100 μL 238 ng/mL GONRs, 100 μL 2.9 mmol/L HAuCl₄, and 50 μL 0.1 mmol/L H₂O₂ solutions were added before diluting to 1.5 mL with water. The mixed solution was placed in a 60 °C water bath for reacting 20 min and the reaction was terminated by tap water. The mixture was transferred into a quartz cell, and its RRS spectra were recorded. The RRS intensity I at 550 nm and the blank I₀ without Na⁺ were recorded, and ΔI = I – I₀ was calculated.

4. Conclusions

Based on the selective reaction of Na⁺ with CAP to control the GONR catalysis of the reduction of HAuCl₄ with H₂O₂ to produce gold nanoparticles with strong RRS effect, a new RRS method for the determination of Na⁺ was established. It has the advantages of simple operation, high sensitivity, and good selectivity.

Data Availability

The data [see 1–23] used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare no conflicts of interest.

Authors’ Contributions

Chongning Li and Haidong Wang acquired data for the work, drafted the work, gave final approval of the version to be published, and agreed to be accountable for all aspects of the work in questions related to its accuracy. Yanghe Luo and Zhiliang Jiang designed the work, analyzed data for the work, revised it critically for important intellectual content, gave final approval of the version to be published, and agreed to be accountable for all aspects of the work in ensuring that integrity of any part of the work was appropriately investigated and resolved.

Acknowledgments

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Supplementary Materials

Fig.S1 RRS spectra of CH₃CH₂OH-Na⁺-PA system. a: 1.13 mol/L CH₃CH₂OH + 3.14 μmol/L CAP; b:a+0.69 mmol/L Na⁺; c:a+4.3 mmol/L Na⁺; d:a+8.6 mmol/L Na⁺; e:a+12.9 mmol/L Na⁺; f:a+17.2 mmol/L Na⁺; g:a+25.8 mmol/L Na⁺. Fig.S2 RRS spectra of CH₃CH₂OH-Na⁺-PA-GO-H₂O₂-HAuCl₄ system. a:1.13 mol/L CH₃CH₂OH+ 3.14 μmol/L CAP+0.193 mmol/L H₂O₂+50 ng/mL GO; b:a+0.86 mmol/L Na⁺; c:a+4.3 mmol/L Na⁺; d:a+8.6 mmol/L Na⁺; e:a+12.9 mmol/L Na⁺; f:a+17.2 mmol/L Na⁺; g:a+25.8 mmol/L Na⁺. Fig.S3 RRS of CH₃CH₂OH-GONR-H₂O₂-HAuCl₄ system. a:1.13 mol/L CH₃CH₂OH+ 3.14 μmol/L CAP+0.193 mmol/L H₂O₂+50 ng/mL GO; b:a+0.79 mmol/L Na⁺; c:a+2.36 μmol/L PA; d:a+3.14 μmol/L PA; e:a+4.0 μmol/L PA; f:a+4.72 μmol/L PA; g:a+5.5 μmol/L PA. Fig.S4 RRS of CH₃CH₂OH-GO-H₂O₂-HAuCl₄ system. a:1.13 mol/L CH₃CH₂OH+ 3.14 μmol/L CAP+0.193 mmol/L H₂O₂+50 ng/mL GO; b:a+0.79 mmol/L Na⁺; c:a+2.36 μmol/L PA; d:a+3.14 μmol/L PA; e:a+4.0 μmol/L PA; f:a+4.72 μmol/L PA; g:a+5.5 μmol/L PA. Fig.S5 RRS spectra of GONR-H₂O₂-HAuCl₄ system. a:1.13 mol/L CH₃CH₂OH +0.193 mmol/L HAuCl₄ +3.3 mmol/L H₂O₂; b:a+1.58 ng/mL GONR; c:a+3.17 ng/mL GONR; d:a+7.93 ng/mL GONR; e:a+11.1 ng/mL GONR; f:a+14.3 ng/mL GONR; g:a+15.8 ng/mL GONR. Fig.S6 RRS spectra of GO-H₂O₂-HAuCl₄ system. a:1.13 mol/L CH₃CH₂OH+0.193 mmol/L H₂O₂+3.3 mmol/L H₂O₂+50 ng/mL GO; b:a+6.6 ng/mL GO; c:a+13.3 ng/mL GO; d:a+16.6 ng/mL GO; e:a+20 ng/mL GO; f:a+26.6 ng/mL GO; g:a+50 ng/mL GO. Fig. S7 UV of CH₃CH₂OH-Na⁺-PA-GO-H₂O₂-HAuCl₄ system. a:1.13 mol/L CH₃CH₂OH+ 3.14 μmol/L CAP+0.193 mmol/L H₂O₂+50 ng/mL GO; b:a+0.86 mmol/L Na⁺; c:a+4.3 mmol/L Na⁺; d:a+12.9 mmol/L Na⁺; e:a+17.2 mmol/L Na⁺; f:a+25.8 mmol/L Na⁺. Fig. S8 UV of CH₃CH₂OH-GONR-H₂O₂-HAuCl₄ system. a:1.13 mol/L CH₃CH₂OH+ 3.14 μmol/L CAP+0.193 mmol/L H₂O₂+50 ng/mL GO; b:a+0.79 mmol/L PA; c:a+2.36 μmol/L PA; d:a+3.14 μmol/L PA; e:a+4.0 μmol/L PA; f:a+4.72 μmol/L PA; g:a+5.5 μmol/L PA. Fig. S9 UV of CH₃CH₂OH-GO-H₂O₂-HAuCl₄ system. a:1.13 mol/L CH₃CH₂OH+ 3.14 μmol/L CAP+0.193 mmol/L H₂O₂+50 ng/mL GO; b:a+0.79 mmol/L PA; c:a+2.36 μmol/L PA; d:a+3.14 μmol/L PA; e:a+4.72 μmol/L PA; f:a+5.5 μmol/L PA; g:a+6.29 μmol/L PA. Fig. S10 UV of CH₃CH₂OH-GONR-H₂O₂-HAuCl₄ system. a:1.13 mol/L CH₃CH₂OH+0.193 mmol/L H₂O₂+3.3 mmol/L H₂O₂; b:a+1.58 ng/mL GONR; c:a+6.34 ng/mL GONR; d:a+9.52 ng/mL GONR; e:a+15.8 ng/mL GONR; f:a+23.8 ng/mL GONR. Fig. S11 UV spectra of CH₃CH₂OH-GO-H₂O₂-HAuCl₄ system. a:1.13 mol/L CH₃CH₂OH+0.193 mmol/L H₂O₂+3.3 mmol/L H₂O₂; b:a+6.6 ng/mL GO; c:a+13.3 ng/mL GO;
Y. Liu and C. Z. Huang, “Screening sensitive nanosensors via Figonr. Na+ + 0.193 mmol/L HAuCl₄ + 3.3 mmol/L H₂O₂. Fig. S12b The effect of PA concentration. 1.13 mol/L CH₃CH₂OH+17.2 mmol/L Na+ + 3.14 μmol/L PA + 0.193 mmol/L HAuCl₄ + 3.3 mmol/L H₂O₂ + 23.8 ng/mL GONR. Fig. S12c The effect of H₂O₂ concentration. 1.13 mol/L CH₃CH₂OH+17.2 mmol/L Na+ + 3.14 μmol/L PA + 0.193 mmol/L HAuCl₄ + 3.3 mmol/L H₂O₂ + 23.8 ng/mL GONR. Fig. S12f The effect of reaction time. 1.13 mol/L CH₃CH₂OH+17.2 mmol/L Na+ + 3.14 μmol/L PA + 0.193 mmol/L HAuCl₄ + 3.3 mmol/L H₂O₂ + 23.8 ng/mL GONR. (Supplementary Materials)

References


