Sensitive Carbon Monoxide Gas Sensor Based on Chemiluminescence on Nano-Au/Nd2O3–Ca3Nd2O6: Working Condition Optimization by Response Surface Methodology

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ABSTRACT: An Au/Nd2O3–Ca3Nd2O6 composite was synthesized by the sol–gel and impregnation method. The EDS spectrum and the transmission electron microscopy image showed that Au atoms are uniformly distributed on the surface of Nd2O3–Ca3Nd2O6 with a size of less than 50 nm. A sensitive carbon monoxide gas sensor based on chemiluminescence at a temperature lower than 200 °C was reported. There is a good linear relationship between the chemiluminescence intensity and the concentration of carbon monoxide in the range of 0.6–125 mg/m³. The detection limit (3σ) is 0.2 mg/m³. The working conditions optimized by the response surface methodology were an analytical wavelength of 620.90 nm, a reaction temperature of 131.63 °C, and a carrier-gas velocity of 105.46 mL/min. The sensitivity of the method can be increased by 4.5% under the optimized working conditions, which is especially important for the determination of trace substances. The carbon monoxide sensor demonstrated in this paper can be used for practical applications. The optimization method is universal for many multiparameter processes.

INTRODUCTION

Carbon monoxide (CO) is a colorless, odorless, and irritant gas. CO is the most widely distributed and the major pollutant in the atmosphere and is also one of the important pollutants generated in the combustion process. There are many methods to determine CO in the laboratory, such as chromatography, fluorescence, colorimetry, infrared spectroscopy, photoacoustic spectroscopy, electrocatalysis, absorption spectrometry, and so on. However, these methods not only need to collect and prepare samples, but also need to use large-scale analytical instruments, so the operation is very complicated. Many new techniques, such as using nanomaterials, mid-infrared, membrane, optical fiber, electrochemistry, oxygen-deficient perovskite, laser, microbial fuel cell, and screen print, have been used to study the rapid determination of CO. Chemiluminescence (CL) on a solid catalyst is usually a heterogeneous catalytic oxidation reaction on a gas–solid interface, which is different from the CL in a solution. It is a promising technology for making gas sensors. In recent years, a series of rapid analytical applications of CL on catalysts have been developed for CO and many other molecules at different laboratories. When using catalysts to research and make a CO gas sensor, high working temperature becomes a main obstacle. Generally, sensors need to be as small or even miniaturized as possible in order to be portable, which implies that they must be a small heat capacity system. We know that it is very easy to control the temperature accurately in the laboratory. However, it is very difficult to maintain the high temperature accuracy and stability in a small heat capacity system, which is totally different from the situation in the laboratory equipment. Our previous work has proposed that the baseline instability will occur in a small heat capacity laboratory. However, it is very difficult to maintain the high temperature accuracy and stability in a small heat capacity system, which is totally different from the situation in the laboratory. Our previous work has proposed that the baseline instability will occur in a small heat capacity system when the working temperature exceeds 200 °C. In the past, the working temperature for CL on catalysts was usually more than 250 °C. These sensitive materials they use have almost no CL activity below 200 °C, so they are not suitable for making CL gas sensors. Au-doped composites often exhibit catalytic oxidation activities at low temperature for CO and many molecules. In addition to Au, other noble metals, such as Pd, Pt, and Ag, show a similar performance. Our team found that Au/Nd2O3–Ca3Nd2O6 shows low-temperature CL activity for CO.

In the past, the working conditions for CL on catalysts were simply determined by single-factor experiments. We know that the premise of single-factor experiments is to assume that there is no interaction between various factors. This
assumption is beneficial to simplify the experiment, but its
defect is obvious. So far, there is no research report about the
interaction of different factors on CL on catalysts, nor the
research of using a statistical model to obtain the optimal
experimental conditions. The response surface method (RSM)
is a statistical comprehensive test technique. It can be
used to analyze the regression relationship between test
indexes (dependent variables) and multiple test factors
(independent variables). It takes the response of the system
as a function of many factors, and carries out the
finite deterministic "trials" on a response surface to simulate the real-
state surface. In this way, we can use intuitive observation to
select the optimal experimental conditions in the experimental
design.

In this work, first, Au-doped Nd$_2$O$_3$–Ca$_3$Nd$_2$O$_6$ was
synthesized and characterized. Then, the experimental
conditions were optimized by the RSM, and the low-
temperature CL properties of the composite were studied.
Finally, a feasible method was established for determining CO
by utilizing CL on solid catalysts at a temperature lower than
200 °C.

### RESULTS AND DISCUSSION

**Characterization of Sensitive Materials.** The EDS
element mapping in Figure 1 shows that oxygen, neodymium,
calcium, and gold were uniformly distributed in the composite.
The EDS spectrum in Figure 2 shows that the prepared
catalyst is Au-doped Nd$_2$O$_3$–Ca$_3$Nd$_2$O$_6$, because the atomic
ratio of O, Nd, and Ca is close to 9:4:3. When the atomic
percentage of Au is less than 0.8%, the composite shows CL
activity to CO only when the temperature is above 200 °C.
When the atomic percentage of Au is more than 1.5%, in
addition to CO, formaldehyde, acetaldehyde, sulfur dioxide,
hydrogen sulfide, and other molecules also show an obvious
CL signal. It can be seen that the content of Au in the catalyst
has a great influence on the selectivity and sensitivity of
catalytic oxidation, and the content of 0.8–1.2% Au in the
catalyst is good for CO catalytic oxidation.

The transmission electron microscopy (TEM) image in
Figure 2 shows that the size of Au/Nd$_2$O$_3$–Ca$_3$Nd$_2$O$_6$ is not
more than 50 nm.

**Factors Influencing the CL Intensity.** In this part, we
simplify the analysis wavelength, reaction temperature, and
carrier-gas velocity as single influencing factors to separately
study their influences on the CL intensity of CO on nano-Au/
Nd$_2$O$_3$–Ca$_3$Nd$_2$O$_6$. 

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**Figure 1.** EDS mapping of Au/Nd$_2$O$_3$–Ca$_3$Nd$_2$O$_6$ (a—test area, b—mix, c—oxygen, d—neodymium, e—calcium, and f—gold).

**Figure 2.** EDS spectrum and the TEM photograph of the prepared catalyst.
Figure 3 is the CL spectrum of 5 mg/m³ CO on nano-Au/Ca₃Nd₄O₉ at 130 °C with a carrier-gas velocity of 100 mL/min. The CL intensities were higher at 620 nm than others. This simple spectral profile suggests that there may be only one luminescent species in the 560–680 nm range, and 620 nm can be used as the primary analysis wavelength.

Figure 4 is the temperature dependence of the CL intensity of 5 mg/m³ CO at a wavelength of 620 nm under a carrier-gas velocity of 100 mL/min. The CL intensity is high near 130 °C.

Figure 5 shows the carrier-gas velocity dependence of the CL intensity of 5 mg/m³ CO at 130 °C. It can be seen from this figure that the carrier-gas velocity of around 100 mL/min deserves attention.

Summing up the above experimental results, it can be concluded that the single-factor experimental conditions are: an analysis wavelength of 620 nm, a reaction temperature of 130 °C, and a carrier-gas velocity of 100 mL/min.

Optimization of Experimental Conditions by the RSM. The analysis wavelength, reaction temperature, and carrier-gas velocity were used as experimental conditions. The CL intensity was used as the response value. According to the experimental conditions preliminarily selected for the single-factor experiment, the Box–Behnken test design of three factors and three levels is shown in Table 1.

The results of 17 response surface design trials (12 edge points plus 5 center points in Box–Behnken test design) for 5 mg/m³ CO are shown in Table 2.

Table 1. Factors and Levels of the Box–Behnken Test Design

| factors | wavelength (nm) | temperature (°C) | flow rate (mL/min) |
|---------|----------------|----------------|-------------------|
| -1      | 610            | 120            | 90                |
| 0       | 620            | 130            | 100               |
| 1       | 630            | 140            | 110               |

Table 2. Response Surface Design Arrangement and the Experimental Results

| number | wavelength (nm) | temperature (°C) | flow rate (mL/min) | CL intensity |
|--------|----------------|----------------|-------------------|--------------|
| 1      | 630.00         | 130.00         | 90.00             | 320          |
| 2      | 620.00         | 120.00         | 90.00             | 231          |
| 3      | 620.00         | 130.00         | 100.00            | 561          |
| 4      | 610.00         | 120.00         | 100.00            | 170          |
| 5      | 630.00         | 130.00         | 110.00            | 495          |
| 6      | 620.00         | 130.00         | 100.00            | 560          |
| 7      | 630.00         | 140.00         | 100.00            | 361          |
| 8      | 620.00         | 130.00         | 100.00            | 559          |
| 9      | 620.00         | 140.00         | 110.00            | 399          |
| 10     | 620.00         | 130.00         | 100.00            | 561          |
| 11     | 620.00         | 130.00         | 100.00            | 559          |
| 12     | 630.00         | 130.00         | 100.00            | 183          |
| 13     | 610.00         | 130.00         | 90.00             | 251          |
| 14     | 610.00         | 140.00         | 100.00            | 320          |
| 15     | 620.00         | 120.00         | 110.00            | 331          |
| 16     | 620.00         | 140.00         | 90.00             | 315          |
| 17     | 610.00         | 130.00         | 110.00            | 438          |

The 3D surfaces and contours are plotted using Design-Expert software 8.0, as shown in Figures 6–8. Each figure represents the interaction of two independent variables with the CL intensity.

Figure 6 is the 3D surface (A) and contours (B) of the interaction of analysis wavelength and reaction temperature with the CL intensity. With the increase of the analysis wavelength and reaction temperature, the CL intensity increases. When the analysis wavelength reaches 620.90 nm and the reaction temperature reaches 131.63 °C, the CL intensity reaches its maximum. When the analysis wavelength
and reaction temperature continue to increase, the CL intensity begins to decrease.

Figure 7 is the 3D surface (A) and contours (B) of the interaction of analysis wavelength and carrier-gas velocity with the CL intensity. As can be seen, when the analytical wavelength reaches 620.90 nm and the carrier-gas velocity reaches 105.46 mL/min, the CL intensity reaches its maximum.
**Figure 8** is the 3D surface (A) and contours (B) of the interaction of reaction temperature and carrier-gas velocity with the CL intensity. As can be seen, when the reaction temperature reaches 131.63 °C and the carrier-gas velocity reaches 105.46 mL/min, the CL intensity reaches its maximum.

Based on the above experimental results, it can be found that the optimum values of analysis wavelength, reaction temperature, and carrier-gas velocity are 620.90 nm, 131.63 °C, and 105.46 mL/min, respectively, when the maximum CL intensity is obtained. It is almost impossible to obtain the optimal operating conditions through single-factor experiments, because the number of experiments needed is very large. According to the model, the maximum CL intensity of 5 mg/m³ CO was 584.5. This is 4.5% higher than the CL intensity under single-factor experimental conditions, which is very important to improve the sensitivity of the method.

**Sensing Properties.** An experiment was carried out to investigate the lifetime of nano-Au/Nd2O3−Ca3Nd2O6 at 131.63 °C by continually introducing 20 mg/m³ CO with a velocity of 105.46 mL/min to the surface of sensitive materials and detecting the CL intensities at 620.90 nm once every hour. The results showed that the relative standard deviation (RSD) of CL intensities was less than 3% for continuous 600 h detection. Further experiments showed that the RSD of the CL intensities was within 5% for daily use above 10 months. It can be said that nano-Au/Nd2O3−Ca3Nd2O6 has a long service life for CO monitoring.

Formaldehyde, acetaldehyde, ammonia, sulfur dioxide, sulfur trioxide, hydrogen sulfide, ethanol, benzene, carbon dioxide, nitrogen dioxide, and methane, which are common molecules in the air, are used as interference molecules to study the CL selectivity of sensitive materials to CO. The CL signals at 620.90 nm of 10 mg/m³ CO and 20 mg/m³ other molecules with a carrier-gas velocity of 105.46 mL/min were investigated on nano-Au/Nd2O3−Ca3Nd2O6 of our choice (1.12% Au) and another (1.80% Au) at 131.63 °C. The results are shown in **Figure 9**.

The CL signals from CO were much larger than from other molecules. The signals from other molecules were less than 2% of CO’s signals. There are almost no CL signals for sulfur trioxide, ethanol, benzene, carbon dioxide, nitrogen dioxide, and methane. It indicated that nano-Au/Nd2O3−Ca3Nd2O6− 1.12% Au had good selectivity for CO. The signals from formaldehyde, acetaldehyde, and ammonia, however, were more than 30% of CO’s signals on another catalyst. Details are shown in **Figure 9**.

**Figure 10** is the regression curve of CL intensity versus CO concentration. As can be seen, it has a good linear relationship in the range 0.6–125 mg/m³ with a detection limit (3σ) of 0.2 mg/m³ under the optimization conditions. The linear equation is \( I = 113.5C + 17.49 \) \( (R^2 = 0.9995) \), where \( I \) is the CL intensity, \( C \) is the concentration of CO and \( R \) is the correlation coefficient.

Response characteristics of CL signals for CO under the optimization conditions were investigated. **Figure 10** shows the dynamic CL response curves for 10 mg/m³ CO. It can be seen that both response and recovery of CO are singularly fast.

**Possible Mechanism.** We have made a preliminary study on the mechanism of this luminescence reaction. The products of catalytic oxidation were analyzed by gas chromatography–mass spectrometry after molecules in air were introduced. When the surface temperature of the catalyst is about 130 °C, only when there is CO in the sample gas could the tail gas change, that is to say, a small amount of carbon dioxide can be detected. There is no change in the other molecules in the sample gas. However, when the catalyst surface temperature is increased to about 180 °C, many molecules in the sample gas can change. For example, a small amount of formaldehyde molecules in the sample gas can be converted into formic acid, a few of sulfur dioxide molecules can be converted into sulfur trioxide, trace of acetaldehyde molecules can be converted into acetic acid, and so forth.

The possible luminescence mechanism is that CO molecules in air can be selectively adsorbed on the active sites of nano-Au/Nd2O3−Ca3Nd2O6 under specific experimental conditions, and then react with pre adsorbed oxygen, therefore, CO is oxidized to carbon dioxide. The light quantum with \( \nu \) wavelength can be emitted when the carbon dioxide molecule on the excited state returns to the ground state. The reaction mechanism can be expressed using the following formula

\[
CO + \frac{1}{2}O_2 \rightarrow CO_2^* \\
CO_2^* \rightarrow CO_2 + h\nu
\]
CONCLUSIONS

A sensitive carbon monoxide gas sensor based on Au-activated Nd$_2$O$_3$−Ca$_3$Nd$_2$O$_6$ at 131.63 °C was demonstrated. Its detection range is 0.6−125 mg/m$^3$, and its detection limit is 0.2 mg/m$^3$.

The atomic percentage of 0.8−1.5% Au in nanocomposites was beneficial in terms of low operating temperature and high selectivity for the CL of carbon monoxide.

The sensitivity of the method can be increased by 4.5% after optimizing the experimental conditions by the response surface methodology.

EXPERIMENTAL SECTION

Chemical Reagents and Apparatus. Calcium nitrate, neodymium oxalate, citric acid, sucrose, ammonia, chloroauric acid, and glucose were purchased from Beijing Chemical Regent Co., Ltd. (Beijing, China). Standard gases of carbon monoxide, formaldehyde, acetdehyde, ammonia, benzene, ethanol, sulfur dioxide, hydrogen sulfide, and carbon dioxide in nitrogen were purchased from Beijing Yanan Gas Co., Ltd. (Beijing, China). Distilled water was used throughout the whole experiment. The micro-area composition and particle morphology of nano-Au/Nd$_2$O$_3$−Ca$_3$Nd$_2$O$_6$ were investigated using a scanning electron microscope (JEOL-IT500) and a TEM (JEOL-2100), respectively. The CL intensities were recorded using an ultraweak luminescence analyzer manufactured at the Biophysics Institute of Chinese Academy of Science (Beijing, China).

Synthesis of Sensing Materials. Calcium nitrate and neodymium oxalate were dissolved in 20% citric acid solution. The solution is stirred using an ultrasonic stirrer to obtain a clear solution. Sucrose was added into the solution in the stirring state, and a sol was obtained by continuously stirring the solution for more than 8 h. The pH value of the solution was adjusted to 4.2 with dilute ammonia water. The solution was stirred for 6 h and aged for 12 h. Then, the gel was prepared by rotating evaporation. After drying and grinding, the gel is placed in a box-type resistance furnace. First, the temperature is increased to 250 °C at a rate of 3 °C/min and maintained for 5 h, then it is increased to 480 °C at the same rate and maintained for 3 h. The composite powder of CaO and Nd$_2$O$_3$ was obtained by natural cooling. The crystals of chloroauric acid and glucose were dissolved in water. In the stirring state, the ultrasonic dispersed composite powder is slowly added into the solution and mixed evenly. After standing, filtering, drying, and heating reduction at 165 °C, another composite powder composed of CaO and Nd$_2$O$_3$ doped with the Au atom was obtained. See Chinese patent for the detailed synthesis process.52 The XRD pattern in Figure 11 shows that it consisted of Au/Nd$_2$O$_3$−Ca$_3$Nd$_2$O$_6$ in which Nd$_2$O$_3$ was tetragonal and Ca$_3$Nd$_2$O$_6$ was monoclinic.

Measurement of Signals of CL on the Solid Catalyst. The CL system used in this work is shown in Figure 12. For details of the loading of nano-Au/Nd$_2$O$_3$−Ca$_3$Nd$_2$O$_6$, the adjustment of working temperature, the introduction of the measured gas sample, and the recording of CL signals, readers can refer to our previous work.52 Experimental Design for RSM Optimization. According to the single-factor experimental results of analysis wavelength, reaction temperature, and carrier-gas velocity, the Box−Behnken central composite experiment was designed, and
the CL experimental conditions of CO were optimized by the RSM at three factors and three levels.

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Notes

The authors declare no competing financial interest.

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