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Determination of bisphenol A based on chemiluminescence from gold(III)–peroxymonocarbonate

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ABSTRACT

Peroxymonocarbonate (HCO_4^-) was produced by the online reaction of bicarbonate with hydrogen peroxide. A strong chemiluminescence (CL) was observed when HCO_4^- reacted with $AuCl_4^-$ without any special CL reagent. When bisphenol A (BPA) was added to $AuCl_4^-$–HCO_4^- CL system, the CL emission was inhibited significantly. This new CL system was developed as a flow-injection method for the determination of BPA. Under the optimum experimental conditions, the inhibited CL intensity was linearly related to the concentration of BPA from 0.3 to 80 μM ($R = 0.9958$). The detection limit of BPA was 0.08 μM. The relative standard deviation for 12 repeated measurements of 1.0 μM BPA was 2.9%. The interferences of some cationic ions can be removed by an online cation-exchange column. The applicability of the present CL system was demonstrated for the sensitive and selective determination of BPA in real samples (mineral water bottle, baby bottle, beverage bottle and polycarbonate container). Based on the CL spectrum, UV–visible adsorption spectra, and the quenching effect of reactive oxygen species scavengers, a possible CL mechanism was proposed.

1. Introduction

Bisphenol A (2,2-bis(4-hydroxyphenyl)propane, BPA) is an important industrial chemical owing to its wide use as an intermediate in the manufacture of polycarbonate plastics, epoxy resins, flame retardants, unsaturated polyester resins, polyacrylate, polyetherimide and polysulphone resins [1,2]. BPA can be released into the environment from a wide variety of food contact materials, such as bottles, packaging, landfill leachates, paper, and plastic plants [3–5]. BPA mainly involves its potential human health risk due to its estrogenic activities. Such estrogenic potencies can interfere with hormonal activities, and thus it has been identified as an important endocrine disrupting compound (EDC). Most importantly, BPA was found to possibly cause various kinds of cancer, pleiotropic actions in the brain and cardiovascular system [6–8]. Therefore, it is necessary to establish an accurate method for the determination of BPA. The classic analytical techniques are currently employed to determine BPA, including fluorescence [9,10], liquid chromatography coupled with mass spectrometry (LC–MS) [11,12], gas chromatography coupled with mass spectrometry (GC–MS) [13,14], enzyme linked immunosorbent assays (ELISA) [15] and electrochemical sensors [16,17].

Chemiluminescence (CL) should be a promising technique with the advantage of reliability, fast response, cheap instrument, low cost, simple operation and high sensitivity [18–20]. To the best of our knowledge, there are only two reports on CL determination for BPA [21,22]. Nakashima and co-workers [21] were the first to demonstrate the feasibility of CL detection for BPA, based on the energy transfer from the reaction intermediate(s) of peroxyoxalate CL reaction to the derivatized BPA. However, the precolumn derivatization steps in this method are time-consuming (20 min) and the excess unreacted derivatization reagent must be removed by a solid-phase extraction procedure; the other major disadvantage of this approach is that peroxyoxalate CL reagents are highly insoluble in aqueous solutions and the CL efficiency can be greatly quenched in the presence of water [23]. Moreover, Wang et al. [22] described a CL method for BPA determination in aqueous media based on the inhibitory effect of BPA on the CL reaction between luminol and potassium hexacyanoferrate, but the detection limit of BPA is only 3.1×10^{-7} M, which is not enough for the trace analysis of BPA in real samples.

It is reported that bicarbonate can react with hydrogen peroxide (H_2O_2) to form unstable peroxymonocarbonate (HCO_4^-) [24], and an ultra-weak CL was observed during the decomposition of HCO_4^- [25]. However, the CL signals generated from HCO_4^- are too weak to detect by the direct CL techniques. Therefore, it is necessary to use some CL enhancement reagents or catalysts to amplify CL

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signals, such as cobalt(II) [26], eosin Y [27] and Eu(II)–dipicolinate complex [28]. In the present work, gold(III) chloride ($AuCl_4^-$) was found to enhance remarkably the CL emission generated from decomposition of HCO_4^-, and BPA can greatly inhibit the CL intensity of $AuCl_4^-$–HCO_4^- system. Based on these phenomena, a new flow-injection CL method, without any special CL reagent, e.g. peroxyoxalate [21] and luminol [22] was developed for the determination of BPA in aqueous solution. To demonstrate the usefulness of the present CL method for the determination of BPA in real samples, the selectivity, recovery, precision, and linear dynamic range were examined. The proposed method was developed for the direct determination of BPA in polycarbonate plastics with satisfactory results. In addition, based on the CL spectrum, UV-visible spectra and the quenching effect of reactive oxygen species, a reasonable CL mechanism from the $AuCl_4^-$–HCO_4^-–BPA system was proposed.

2. Experimental

2.1. Reagents

All reagents were of analytical grade and used without further purification. Hydrogen tetrachloroaurate(III) trihydrate ($HAuCl_4 \cdot 3H_2O$) was purchased from Acros. The working solutions of $HAuCl_4$ were freshly prepared by diluting 5% $HAuCl_4$ stock solution with ultrapure water. 0.008 M H_2O_2 was freshly prepared by volumetric dilution of commercial 30% (v/v) H_2O_2 (Beijing Chemical Reagent Company) with ultrapure water. A 0.3 M $NaHCO_3$ working solution was prepared by dissolving appropriate amounts of $NaHCO_3$ (Beijing Chemical Reagent Company) in ultrapure water. 0.1 M BPA stock solution was prepared by 0.2283 g BPA (Tanjin Chemical Reagent Company) in 10 mL anhydrous ethanol (Beijing Chemical Reagent Company), and its working standard solutions were freshly prepared by diluting the stock solution with ultrapure water. The cation-exchange resin (732#) was purchased from Tianjin Chemical Reagent Company. 1,4-Diazabicyclo[2.2.2]octane (DABCO), 36% (v/v) HCl, ascorbic acid and NaN_3 were from Tanjin Chemical Reagent Company.

2.2. Apparatus

UV–visible spectra were measured on a USB 4000 miniature fiber optic spectrometer in absorbance mode with a DH-2000 deuterium and tungsten halogen light source (Ocean Optics, Dunedin, FL). Gold nanoparticles were confirmed through transmission electron microscope (TEM) using a HITACHI-800 (Hitachi, Tokyo, Japan). Emission fluorescence spectra were obtained with a Hitachi F7000 spectrofluorimeter equipped with FL Data Manager Software for spectral acquisition. The emission and excitation slit was maintained at 5 nm. The scan rate of the monochromators was maintained at 240 nm/min. The excitation wavelength was set at 278 nm. The CL detection was conducted on a BPCL luminescence analyzer (Institute of Biophysics, Chinese Academy of Science, Beijing, China). The CL spectra of this system were measured with high-energy cutoff filters from 400 to 640 nm between the flow CL cell and the photomultiplier tube.

2.3. Procedures

A flow system was illustrated in Fig. 1. $HAuCl_4$ solution was injected into the carrier stream (water or sample solution, 1.5 mL/min) through a valve injector with a 120 μL sample loop, and then mixed with HCO_4^-, which was produced online by the reaction between bicarbonate and H_2O_2. The solutions of H_2O_2 and bicarbonate were pumped by the peristaltic pump at 1.3 mL/min. The CL signals generated from the CL cell were detected and amplified by photomultiplier tube. The signal was imported to the computer for data acquisition. The experimental parameters and concentration of sample were measured by the decrease in the CL intensity ($\Delta I = I_0 - I$, where I_0 stands for the signal in the absence of BPA and I stands for the signal in the presence of BPA).

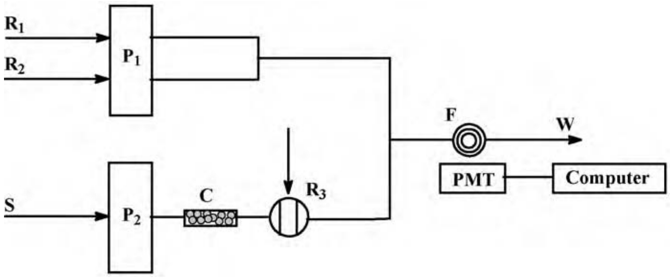


Fig. 1. Schematic diagram of the flow-injection CL detection system. R_1, 0.008 M H_2O_2; R_2, 0.3 M $NaHCO_3$; H_2O_2 and $NaHCO_3$ solution at 1.3 mL/min. S, carrier (water or BPA) at 1.5 mL/min. P_1 and P_2, peristaltic pump; R_3, 120-μL mixed solution of 80 μM $HAuCl_4$ (pH 1.35); C, 3.0 mm i.d. × 15.0 cm length column packed with 732# cation-exchange resin; F, flow cell; W, waste; PMT, photomutiplier tube (−1000 V).

2.4. Sample pretreatment

A baby bottle, beverage bottle, polycarbonate container and mineral water bottles made from polycarbonate plastic were pretreated as the methods described elsewhere [21,22] with minor modification. In brief, the samples were cut into small pieces (size ~$0.5\,cm^2$), and then an accurately weighed 10 g sample was brought to a vigorous boil in a round-bottom 100 mL flask fitted with a reflux condenser. The solution was maintained at the boiling point for 30 min. After the solution was cooled to room temperature, the content of BPA in the aqueous extracts was determined by the proposed method.

3. Results and discussion

3.1. HCO_4^- preparation

It is well-known that HCO_4^- is unstable and difficult to store [29]. Therefore, in the present system, HCO_4^- solution was prepared by the online mixing H_2O_2 and bicarbonate through the flow-injection system. The mixing time interval of H_2O_2 and bicarbonate was controlled by their flow rates. The effect of flow rate for H_2O_2 and bicarbonate on the CL reaction was examined in the range of 0.7–1.7 mL/min. It was observed that the relative CL signal was increased with the increase of flow rate from 0.7 to 1.3 mL/min, however, it was decreased over 1.3 mL/min. Therefore, the optimum flow rate for H_2O_2 and bicarbonate was 1.3 mL/min.

3.2. Concentration and pH of $HAuCl_4$

The effect of $HAuCl_4$ concentration on the relative CL intensity (ΔI) was examined in the range of 1×10^{-5} to 2.5×10^{-4} M. The CL intensities were increased with increasing $HAuCl_4$ concentration up to 8×10^{-5} M, and the CL signals of the higher concentrations than 8×10^{-5} M remained on a plateau. A $HAuCl_4$ concentration of 8×10^{-5} M was chosen as the optimum condition throughout this study on account of its cost.

It was recently reported that Au(III) has five pH-dependent complexes, $AuCl_4^-$, $AuCl_3(OH)^-$, $AuCl_2(OH)_2^-$, $AuCl(OH)_3^-$ and $Au(OH)_4^-$. Among them, $AuCl_4^-$ ions have more reactive activity than the other Au(III) complexes [30,31]. In this work, the pH of $HAuCl_4$ solution was investigated in the range of 1.2–1.9 by the addition of HCl. As shown in Fig. 2, when the pH of $HAuCl_4$ solution is below 1.35, the strongest CL signals can be recorded, which

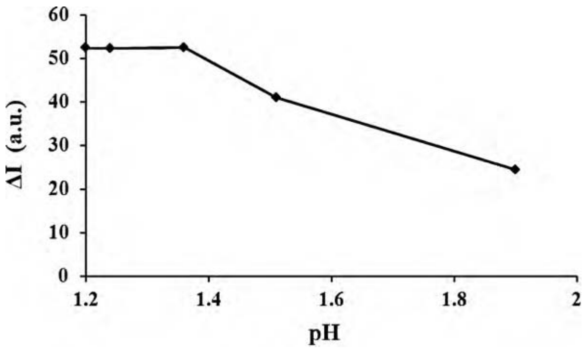


Fig. 2. Effect of pH of $HAuCl_4$ solution on the inhibited CL intensity ($\Delta I = I_0 - I$, where I_0 and I were the CL signals in the absence and in the presence of BPA, respectively) of 5 μM BPA. Other experimental conditions are same as those described in Fig. 1.

showed the $AuCl_4^-$ species played a key role in the increase of CL signals. Therefore, the pH of 1.35 was chosen as the optimum one for the following experiments.

3.3. Concentration of bicarbonate

The CL intensity was strongly dependent on the concentration of bicarbonate. As shown in Fig. 3, in the absence of bicarbonate, there was no CL emission observed, when the concentration of bicarbonate was 0.3 M, the CL emission was the strongest. The concentrations of bicarbonate higher than 0.3 M caused low CL intensity. This phenomenon can be explained that the excess bicarbonate resulted in higher pH; however, as shown in Fig. 2, the higher pH can decrease the ratio of the species of $AuCl_4^-$ complexes, and finally lead to the lower CL signal [30]. Therefore, this system was operated at 0.3 M bicarbonate.

3.4. Concentration of hydrogen peroxide

The effect of the concentration of H_2O_2 on the CL intensity was investigated. Without H_2O_2 in the reaction, no CL signals were observed. As shown in Fig. 4, when the concentration of H_2O_2 is 0.008 M, the most suitable CL emission can be recorded. The concentrations of H_2O_2 higher than 0.008 M caused low CL intensity. This is presumably due to the fact that the higher concentrations of H_2O_2 could reduce $AuCl_4^-$ to Au, resulting in the decrease of $AuCl_4^-$ content in the experimental solution [32,33].

3.5. Analytical performances

Under the optimum experimental conditions employed in the present study, the calibration curve was found to be linear from 0.3 to 80 μM for BPA (Fig. 5), and a regression line $Y = 17.069\ln(X) + 22.665$ ($R = 0.9958$), where Y is the relative CL intensity ($\Delta I = I_0 - I$) and X is the concentration of BPA (Fig. 5, inset). The detection limit (S/N = 3) was 0.08 μM BPA. The relative standard deviation for 12 repeated measurements of 1.0 μM BPA was 2.9%.

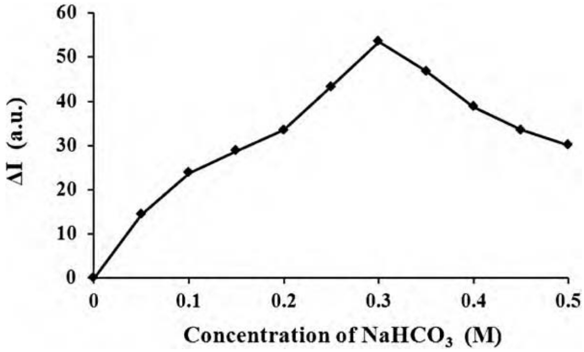


Fig. 3. Effect of $NaHCO_3$ concentration on the inhibited CL intensity of 5 μM BPA. Other experimental conditions are same as those described in Fig. 1.

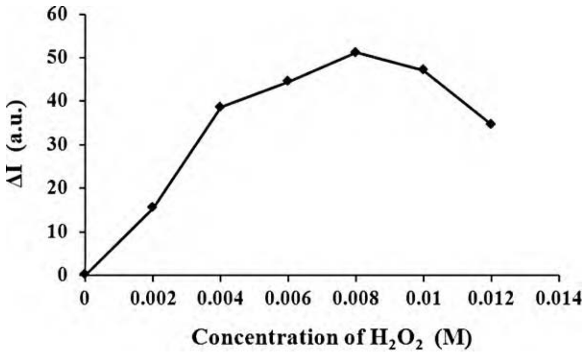


Fig. 4. Effect of hydrogen peroxide concentration on the inhibited CL intensity of 5 μM BPA. Other experimental conditions are same as those described in Fig. 1.

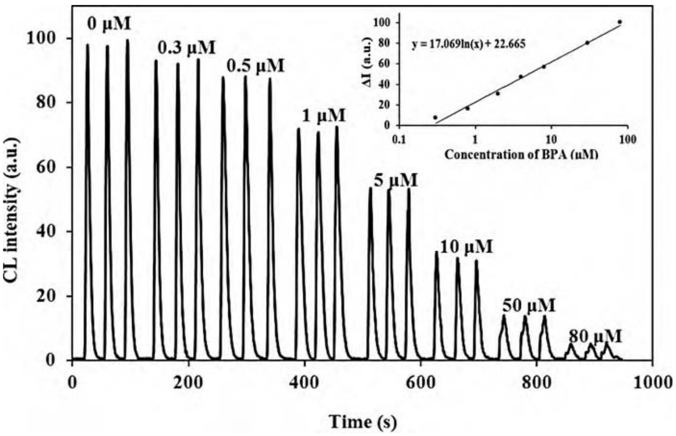


Fig. 5. The CL inhibition of $AuCl_4^-$–HCO_4^- system by adding different concentration of BPA. Inset, the calibration curve for detection of BPA.

3.6. Interference

To assess the selectivity of the developed method, the effects of typical commonly interferences present in mineral water bottle samples were investigated. The tolerance limit was taken as the amount which caused an error of less than 5% for the determination of 1.0 μM BPA. The results were summarized in Table 1. The influences of anionic ions on the CL intensity were slight, most cationic ions, 500-fold K^+, Ca^{2+}, Mg^{2+}, Zn^{2+}, Pb^{2+}, and 50-fold Ba^{2+}, Al^{3+}, have no influence on the determination of 1.0 μM BPA. However, few cationic ions, such as Co^{2+} and Cu^{2+}, interfered seriously. In order to eliminate the interferences from these cationic ions, a column packed with 732# cation-exchange resin with glass wool at both ends (glass tube: 15.0 cm in length and 3 mm i.d.) was assembled

Table 1
The tolerable concentration ratios of some interfering species to 1 μM BPA.

Tolerance (M)	Coexistent substances
5.0×10^{-4}	K^+, Na^+, Ca^{2+}, Mg^{2+}, Pb^{2+}, NH_4^+, NO_3^-, SO_4^{2-}, HPO_4^-,
5.0×10^{-5}	Ba^{2+}, Al^{3+}
5.0×10^{-6}	Fe^{2+}, Zn^{2+}, Ni^{2+}
9.0×10^{-8}	Fe^{3+}, Mn^{2+}, Cr^{3+}
1.0×10^{-13}	Cu^{2+}, Co^{2+}

Table 2
BPA amount and recovery in real samples.

Samples	Measured[a] (μM)	BPA concentration (μM)		Recovery (%)
		Added	Found[a]	
1	0.06 ± 0.003	0.1	0.10 ± 0.004	100 ± 4.0
		0.2	0.20 ± 0.003	100 ± 1.5
2	0.13 ± 0.007	0.25	0.24 ± 0.004	96 ± 1.6
		0.5	0.49 ± 0.002	98 ± 0.8
3	0.12 ± 0.004	0.25	0.26 ± 0.007	104 ± 2.8
		0.5	0.50 ± 0.005	100 ± 1.0
4	0.15 ± 0.005	0.25	0.26 ± 0.004	103 ± 1.6
		0.5	0.49 ± 0.006	98 ± 1.2
5	0.17 ± 0.008	0.25	0.27 ± 0.005	108 ± 2.0
		0.5	0.51 ± 0.003	102 ± 0.6
6	0.09 ± 0.007	0.25	0.26 ± 0.002	104 ± 0.8
		0.5	0.49 ± 0.003	98 ± 0.6

[a] Mean ± SD of three measurements; sample 1: baby bottle; sample 2: beverage bottle; sample 3: polycarbonate container; sample 4: Master Kong mineral water bottle; sample 5: Wahaha mineral water bottle; sample 6: ice dew mineral water bottle.

after the sample flow. The results showed that the interferences of these cationic ions can be removed well.

3.7. Analysis of real samples

In order to evaluate the applicability and reliability of the proposed methodology, it was applied to the determination of BPA in real samples, such as mineral water bottle, baby bottle, beverage bottle and polycarbonate container. We noticed that the pH of sample solution was almost the same as the one of deionized water, and thus the sample solution was pumped directly into the present flow-inject analysis for determination of BPA. Using the standard addition method, the calibration curve in real samples was obtained and used to determine the BPA (Table 2). These results are in concordance with those previously reported [34,35]. The recoveries for BPA in spiked samples were found to be between 98% and 108%.

3.8. Possible mechanism of the present CL system

3.8.1. Emitting species

Cui et al. [36] investigated that the Au(I) complexes were formation by the oxidation reaction of the surface gold atoms with $CO_3^{\bullet -}$, which were emissive in the range of 430–450 nm. Herein, the CL spectrum of $AuCl_4^-$–HCO_4^- system was examined by a series of high-energy cutoff filters from 400 to 640 nm (Fig. 6). It was clearly indicated that there was only one emission band in the range of 430–450 nm, which was probably attributed to such gold(I) intermediates [36].

The emitting species from the examined system was also confirmed by the quenching effect of reactive oxygen species. 1,4-Diazabicyclo[2.2.2]octane (DABCO) and NaN_3, well-known quenchers of singlet oxygen ($^1O_2$) [37], were used in the present experiment, and the results showed that 50 mM DABCO or NaN_3 did not decrease the CL intensity, which provided strong evidence that $^1O_2$ did not contribute to the observed CL. However, the CL signal intensity was effectively quenched by 0.2 mM ascorbic acid (quencher of hydroxyl radical, $HO^\bullet$) [38], which was a strong indication that $HO^\bullet$ was one of intermediates.

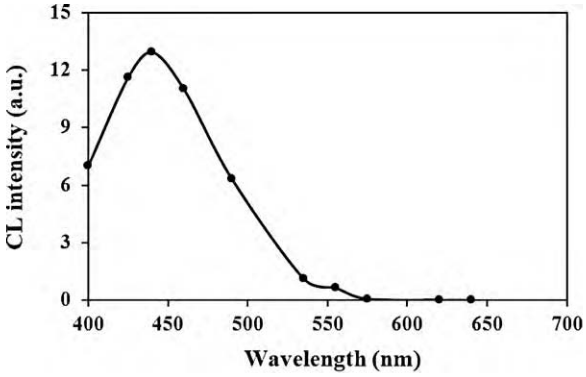


Fig. 6. The CL spectra of $AuCl_4^-$–HCO_4^- system.

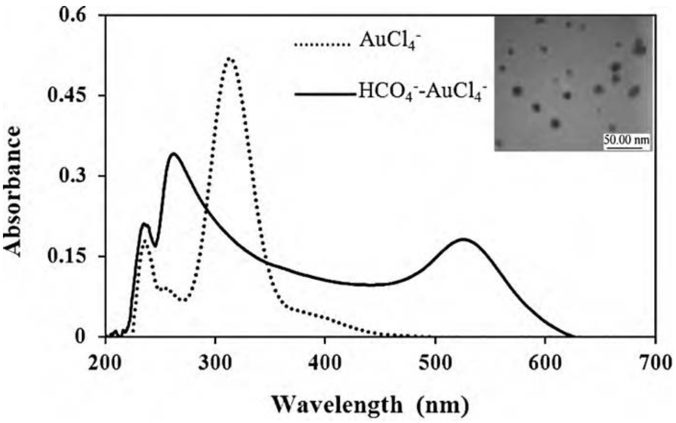


Fig. 7. UV–visible absorption spectra for $AuCl_4^-$ in the absence and presence of HCO_4^-. Inset, TEM image of gold nanoparticles produced by the reaction of $AuCl_4^-$ and HCO_4^-.

3.8.2. UV–visible spectra

As shown in Fig. 7, a sharp absorption peak at 320 nm is the characteristic band of $AuCl_4^-$ [31]. However, a rapid spectrum change was observed upon the addition of HCO_4^- into the $AuCl_4^-$ solution. Interestingly, a new absorption band at around 525 nm appeared in the spectrum, indicating the formation of gold nanoparticles [39]. We verified the formation of gold nanoparticles using the TEM image and the average diameter calculated from the TEM micrographs was 10 ± 2.0 nm (Fig. 7, inset).

3.8.3. Fluorescence spectra

The fluorescence spectra of BPA solution were presented in Fig. 8. When $HAuCl_4$, HCO_4^- or $AuCl_4^-$–HCO_4^- was added separately to the BPA solution, the fluorescence intensity at 307 nm was decreased. Noticeably, the decrease of fluorescence intensity in the presence of HCO_4^- was almost same as $AuCl_4^-$–HCO_4^-. These results proved that the decomposition intermediate ($CO_3^{\bullet -}$ and $HO^\bullet$) of HCO_4^- mainly contributed to the degradation of BPA [25].

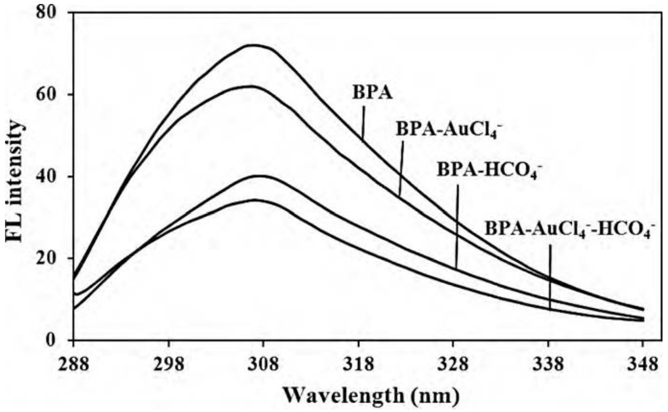
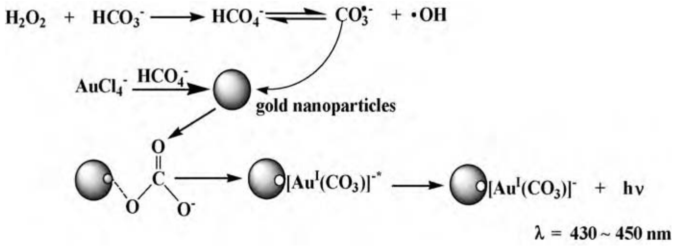


Fig. 8. The emission spectra of BPA, BPA–$HAuCl_4$, BPA–HCO_4^-, BPA–$AuCl_4^-$–HCO_4^-, respectively, recorded at λ_{ex} = 278 nm. Conditions: 5 μM BPA, 80 μM $HAuCl_4$, 0.008 M H_2O_2 and 0.3 M $NaHCO_3$.



Scheme 1. CL reaction mechanism for H_2O_2–$NaHCO_3$–$AuCl_4^-$ system.

These results were in good agreement with the reported studies [40,41].

Based on the above results, the CL mechanism of the present system can be summarized as the follow reactions: HCO_4^- was formed by the reaction of bicarbonate with H_2O_2 (Reaction (1)). It has been proposed [25] that HCO_4^- can rapidly decompose into $CO_3^{\bullet-}$ and $HO^{\bullet}$ by the O–O bond homolysis (Reaction (2)).

$$H_2O_2 + HCO_3^- \rightleftharpoons HCO_4^- \tag{1}$$

$$HCO_4^- \rightarrow HO^{\bullet} + CO_3^{\bullet-} \tag{2}$$

As spectrophotometrically experimental data and TEM image were shown in Fig. 7, the rapid reaction of HCO_4^- with $AuCl_4^-$ resulted in the formation of gold nanoparticles [39] (Reaction (3)).

$$HCO_4^- + AuCl_4^- \rightarrow \text{nanogold} \tag{3}$$

The oxidation of the surface gold atoms by $CO_3^{\bullet-}$ could generate CL by the formation of Au(I) complexes and the emission band is approximately at 430–450 nm [36], which fitted with the experimental CL spectrum shown in Fig. 6 (Reaction (4)).

$$Au_n + CO_3^{\bullet-} \rightarrow Au_{n-1}[Au^I(CO_3)]^-* \rightarrow Au_{n-1}[Au^I(CO_3)]^- + h\nu \tag{4}$$

To obtain further insight into the mechanism of the CL system, the above reactions were summarized in Scheme 1. Gold nanoparticles can be generated immediately when $AuCl_4^-$ solution was injected into the mixed solution of H_2O_2 and $NaHCO_3$ (Fig. 7). HCO_4^- can rapidly decompose into $CO_3^{\bullet-}$ and $HO^{\bullet}$ by the O–O bond hemolysis. $CO_3^{\bullet-}$ can absorb on the surface of gold nanoparticles and oxidize the surface gold atoms to form gold(I) complexes intermediates. These Au(I) complexes intermediates can emit light at 430–450 nm [34].

When BPA solution was added to $AuCl_4^-$–HCO_4^- system, the CL intensity from $AuCl_4^-$–HCO_4^- system decreased dramatically. We speculated that the degradation of BPA by the reaction with the decomposition intermediate of HCO_4^- can compete with $AuCl_4^-$, resulting in the decrease of HCO_4^- content [38,39].

4. Conclusions

In this paper, a new $AuCl_4^-$–HCO_4^- CL system was developed and a possible CL mechanism was proposed by the CL spectrum, UV–visible adsorption spectra, and the quenching effect of reactive oxygen species. Finally, it was successfully applied to the determination of BPA in real samples. When compared to the CL analytical methods for the detection of BPA, which employed organic peroxyoxalate and luminol systems, the present study overcame the weak solubility of peroxyoxalate CL in aqueous solution and exhibited higher sensitivity and tolerance towards interferences. Moreover, we found that HCO_4^- is easy to decompose BPA, which is of great potential for exploring the new green degradation route to the endocrine disrupting compounds.

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