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Chemiluminescence determination of surfactant Triton X-100 in environmental water with luminol-hydrogen peroxide system

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Abstract

Background: The rapid, simple determination of surfactants in environmental samples is essential because of the extensive use and its potential as contaminants. We describe a simple, rapid chemiluminescence method for the direct determination of the non-ionic surfactant Triton X-100 (polyethylene glycol tert-octylphenyl ether) in environmental water samples. The optimized experimental conditions were selected, and the mechanism of the Luminol-H₂O₂-Triton X-100 chemiluminescence system was also studied.

Results: The novel chemiluminescence method for the determination of non-ionic surfactant Triton X-100 was based on the phenomenon that Triton X-100 greatly enhanced the CL signal of the luminol-H₂O₂ system. The alkaline medium of luminol and the pH value obviously affected the results. Luminol concentration and hydrogen peroxide concentration also affected the results. The optimal conditions were: Na₂CO₃ being the medium, pH value 12.5, luminol concentration 1.0×10^{-4} mol L⁻¹, H₂O₂ concentration 0.4 mol L⁻¹. The possible mechanism was studied and proposed.

Conclusion: Under the optimal conditions, the standard curve was drawn up and quotas were evaluated. The linear range was 2×10^{-4} g·mL⁻¹– 4×10^{-2} g·mL⁻¹ (w/v), and the detection limit was 3.97×10^{-5} g·mL⁻¹ Triton X-100 (w/v). The relative standard deviation was less than 4.73% for 2×10^{-2} g·mL⁻¹ (w/v) Triton X-100 (n = 7). This method has been applied to the determination of Triton X-100 in environmental water samples. The desirable recovery ratio was between 96%–102% and the relative standard deviation was 2.5%–3.3%. The luminescence mechanism was also discussed in detail based on the fluorescence spectrum and the kinetic curve, and demonstrated that Triton X-100-luminol-H₂O₂ was a rapid reaction.

Background

Surfactants are employed in a vast number of uses including domestic and industrial detergents, solubilization of membranes, and pharmaceutical and cosmetic formulations. Until recently, surfactants have drawn much research interest as enhancers in some special reactions [1,2]. With these extensive uses (more than 15 million tonnes/year), their toxicity and negative effect on the self-purification capability of surface water, make these syn-

thetic organic compounds one of the main environmental concerns. Therefore, a number of sensitive and high resolution techniques have been developed for the determination of low concentrations of cationic surfactants and anionic surfactants in different matrices, including gas and liquid chromatography [3-7], spectrophotometry [8-11], electrochemical methods [12,13], capillary electrophoresis [14,15] and sensors [16,17]. Chromatographic techniques are the major methods in the analysis of sur-

factants due to the high efficiency and high sensitivity. However, the most important drawback for practical applications is their complication, and chromatographic techniques do not lend themselves to rapid analysis or to on-site studies. Moreover, there are few assays for the determination of non-ionic surfactants.

In recent years, extremely sensitive analytical techniques based on chemiluminescence (CL) and bioluminescence systems have received considerable attention. Simplicity of detection, low detection limits, large calibration ranges and short analysis times are some of the characteristics that make the methods attractive. Additionally, there are a few reports about using CL methods to analyze surfactants in environmental samples [18,19].

TritonX-100 is a water-soluble, liquid, non-ionic surfactant that is recognized as the performance standard among similar products. It is an octylphenol ethoxylate with an average of nine to ten moles of ethylene oxide and is a 100-percent active product. It is widely used as a substrate for detergents and in textile and fiber manufacture because of its excellent detergency, excellent wetting ability and excellent grease and oil removal from hard surfaces[20]. In addition, it is also used as an enhancer in some reactions [21-23]. However, the detection methods for Triton X-100 are limited [24,25]. To our knowledge, up to now, CL determination of Triton X-100 has not been reported.

The aim of our study is to develop a simple, rapid and sensitive CL method for the determination of non-ionic surfactant Triton X-100. This method is based on the fact that Triton X-100 could enhance the CL emission arising from the reaction of luminol with hydrogen peroxide. It has been successfully used to determine Triton X-100 in environmental water samples. Moreover, the mechanism of the Luminol- H_2O_2 -Triton X-100 chemiluminescence system was studied in detail.

Results and discussion

Optimization of alkaline medium

It is well known that the luminol-hydrogen peroxide CL system requires an alkaline medium. Our preliminary experiments showed that the CL signals produced from the reaction of Triton X-100, luminol and H_2O_2 changed greatly with the different alkaline medium in an ultra-weak luminescence analyzer. So the effect of different alkaline mediums with the same pH value at 11 ($\text{NH}_3 \cdot \text{H}_2\text{O}$, NaOH, NaHCO_3 , Na_2CO_3 ; $\text{NaHCO}_3 = 1:3$, Na_2CO_3 ; $\text{NaHCO}_3 = 2:3$, Na_2CO_3 ; $\text{NaHCO}_3 = 1:1$, Na_2CO_3 ; $\text{NaHCO}_3 = 3:2$, Na_2CO_3 ; $\text{NaHCO}_3 = 3:1$) on the CL intensity was investigated. The concentration of NaHCO_3 and Na_2CO_3 was $0.2 \text{ mol} \cdot \text{L}^{-1}$. NaOH concentration was $0.001 \text{ mol} \cdot \text{L}^{-1}$ and $\text{NH}_3 \cdot \text{H}_2\text{O}$ concentration was $0.1 \text{ mol} \cdot \text{L}^{-1}$. The results (Fig. 1) showed that when the proportion of Na_2CO_3 and NaHCO_3 was 3:1, the maximum CL signal was obtained. Nevertheless, the blank signal of the system was too strong. So $0.2 \text{ mol} \cdot \text{L}^{-1} \text{Na}_2\text{CO}_3$ was chosen because the CL signal was much stronger than

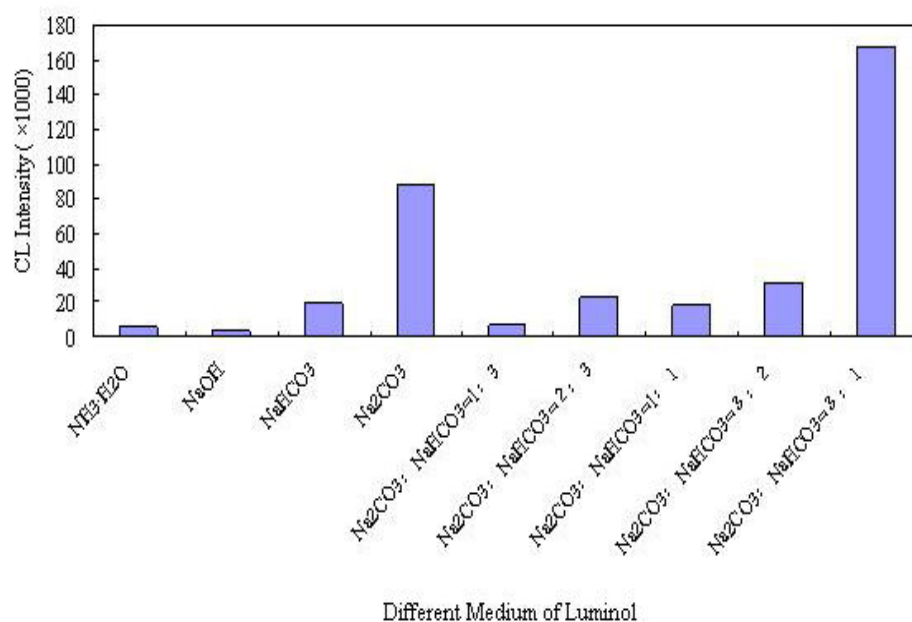


Figure 1
Effect of different medium of luminol on the CL intensity.

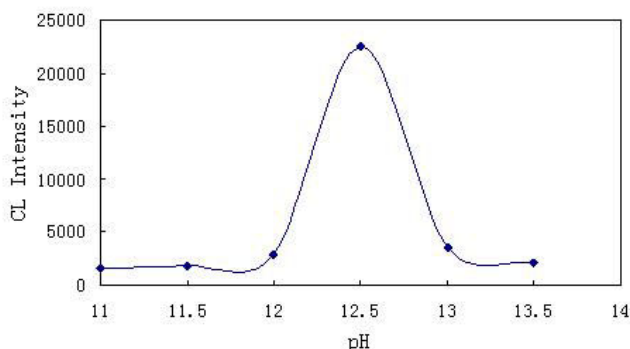


Figure 2
Effect of pH on the CL intensity.

other mediums and the best signal-to-noise ratio was obtained.

Effect of pH of luminol

The effects of pH (11–13.5) were also investigated. The results (Fig. 2) showed that ΔI reached the maximum value when the pH was 12.5, above which the CL intensity decreased. For obtaining better signal-to-noise ratios, pH 12.5 of luminol solution was selected as optimum.

Effect of luminol concentration

As the chemiluminescence reagent, luminol concentration affected the CL intensity. The experimental results showed that the CL intensity increased from $1.0 \times 10^{-5} \text{ mol} \cdot \text{L}^{-1}$ to $2.0 \times 10^{-3} \text{ mol} \cdot \text{L}^{-1}$ and reached the maximum value at the luminol concentration of $1.0 \times 10^{-4} \text{ mol} \cdot \text{L}^{-1}$. So $1.0 \times 10^{-4} \text{ mol} \cdot \text{L}^{-1}$ was chosen as the optimal luminol concentration for subsequent experiments.

Effect of hydrogen peroxide concentration

The effect of H_2O_2 concentration ($0.05 \text{ mol} \cdot \text{L}^{-1}$ – $0.5 \text{ mol} \cdot \text{L}^{-1}$) on the CL intensity was examined. The best signal-to-noise ratio was achieved when the concentration of H_2O_2 was $0.4 \text{ mol} \cdot \text{L}^{-1}$. So $0.4 \text{ mol} \cdot \text{L}^{-1}$ was chosen as the optimal H_2O_2 concentration.

The analytical characteristics of the CL method

Under the optimum experimental conditions, CL response to Triton X-100 concentration was linear in the range of $2 \times 10^{-4} \text{ g} \cdot \text{mL}^{-1}$ – $4 \times 10^{-2} \text{ g} \cdot \text{mL}^{-1}$ (w/v). The detection limit was $3.97 \times 10^{-5} \text{ g} \cdot \text{mL}^{-1}$ (w/v), calculated from

the International Union of Pure and Applied Chemistry (IUPAC) recommendations (3σ). The regression equation was $\Delta I = 474303C - 35.775$ with a correlation coefficient of 0.9996. The relative standard deviations (RSD) for 7 injections with $2 \times 10^{-2} \text{ g} \cdot \text{mL}^{-1}$ (w/v) Triton X-100 was 4.73%.

Interference

Several research works have confirmed that some metal ions (Fe^{2+} , Co^{2+} and Cu^{2+}) could catalyze and enhance the CL signals greatly in a luminol- H_2O_2 CL system [26,27]. Therefore, it is necessary to examine the interference of some coexisting foreign inorganic ions and organic compounds on the luminol- H_2O_2 -Triton X-100 system in order to evaluate the selectivity of the new method. The interference studies were conducted by analyzing a standard solution of $1.0 \times 10^{-2} \text{ g} \cdot \text{mL}^{-1}$ TritonX-100 to which increasing amounts of foreign ions are added. The tolerated limit of each foreign species was taken as a relative error of $\pm 5\%$. The tolerable ratio for foreign species was 1-fold for PO_4^{3-} , 0.1-fold for Na^+ , Cl^- , 0.1-fold for K^+ , 1×10^{-4} -fold for Pb^{2+} , Cu^{2+} , 1×10^{-9} -fold for Ni^{2+} , 1×10^{-10} -fold for Co^{2+} , 0.5-fold for glucose, starch, respectively. Moreover, three kinds of other surfactant CTMAB, SDBS and Tween-80, which may co-exist with the Triton X-100 in the water sample were investigated. The tolerable ratio was 5-fold for CTMAB and Tween-80, 2-fold for SDBS. It can be seen that severe interference was caused by Co^{2+} and Ni^{2+} . Thus, $1.0 \times 10^{-3} \text{ mol} \cdot \text{L}^{-1}$ of EDTA was added to the luminol solution to restrain the interference from metal ions. With the addition of EDTA, the tolerable ratio improved to 1-fold for Ni^{2+} , 1×10^{-4} -fold for Co^{2+} . It demonstrated that EDTA had an obvious restraining effect on metal ions.

Sample analysis

The application of the proposed method was performed by testing environmental water and the recovery of known amounts of standard solution of Triton X-100 added to the different samples. The results obtained are shown in Table 1. As can be seen, the recoveries of added Triton X-100 were quantitative and the reproducibility was satisfactory.

Kinetic curve of the CL reaction

The kinetic curve of the CL reaction in the presence and absence of Triton X-100 was investigated with a static method, using the procedure shown in the experimental

Table 1: Determination and recovery of Triton X-100 in lake water samples

| Samples | Triton X-100 residue | Added(w/v) $\text{g} \cdot \text{mL}^{-1}$ | Found(w/v) $\text{g} \cdot \text{mL}^{-1}$ | Recovery(%) | R.S.D(%) (n = 7) |
|--------------------|----------------------|---|---|-------------|---------------------|
| Nanhu Lake water | -- | 5×10^{-3} | 4.8×10^{-3} | 96 | 2.5 |
| Tangxun Lake water | -- | 5×10^{-3} | 5.1×10^{-3} | 102 | 3.3 |

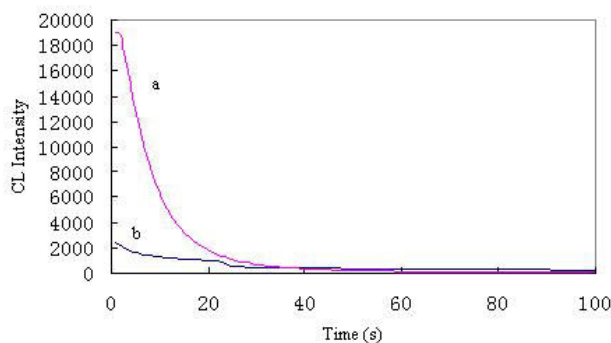


Figure 3
The kinetic curve of the Triton X-100-luminol-H₂O₂ CL system and luminol-H₂O₂ CL system.

section. The results (Fig. 3) showed that it was a fast-type luminescence and the CL intensity reached a maximum within 2 s after the addition of Triton X-100. Comparing curve a (in the presence of Triton X-100) to curve b (in the absence of Triton X-100), we could draw a conclusion that Triton X-100 could enhance the CL reaction in the luminol-H₂O₂ system because the CL signal of curve a (Triton X-100-luminol-H₂O₂) was much stronger than the CL signal of curve b. The kinetic curve indicated the CL method was rapid and suitable to perform a determination of Triton X-100.

The CL reaction mechanism

Several research works were focused on the CL reaction mechanism of luminol. The research results have confirmed that the 3-aminophthalate anion is the emitter in the luminol CL system no matter which medium and oxidant were being used [28,29]. The CL emission of this reaction was generated from the 3-aminophthalate anion rebounding to the ground state from the excited state. The maximum emission was 425 nm. In order to confirm which was the emitter in the Triton X-100-luminol-H₂O₂ system, the fluorescence spectrum (Fig. 4) was measured by spectrophotometer. From Fig. 4, the results indicated

Table 2: The difference of CL signal in the presence and absence of Triton X-100 in luminol-H₂O₂ system

| CL System | CL Signal |
|---|-----------|
| luminol-H ₂ O ₂ | 2831 |
| luminol-H ₂ O ₂ -Triton X-100 (0.05%) | 3085 |
| luminol-H ₂ O ₂ -Triton X-100 (0.1%) | 3333 |
| luminol-H ₂ O ₂ -Triton X-100 (0.2%) | 3774 |
| luminol-H ₂ O ₂ -Triton X-100 (0.5%) | 5704 |
| luminol-H ₂ O ₂ -Triton X-100 (1%) | 7644 |
| luminol-H ₂ O ₂ -Triton X-100 (2%) | 12312 |
| luminol-H ₂ O ₂ -Triton X-100 (4%) | 18874 |
| luminol-H ₂ O ₂ -Triton X-100 (8%) | 26776 |

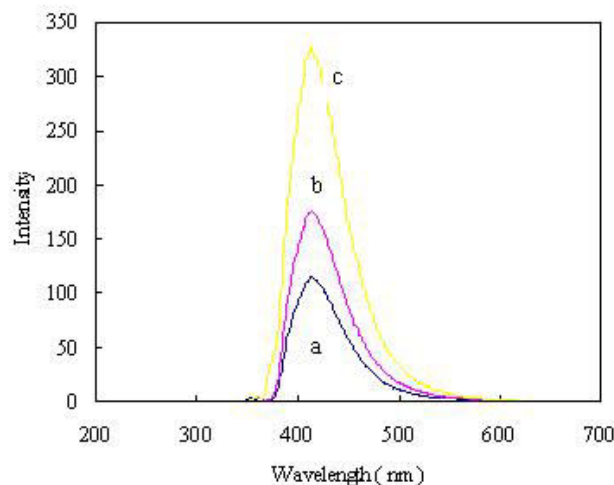


Figure 4
Fluorescence Spectrum of three systems. a. luminol solution b. luminol-H₂O₂ c. luminol-H₂O₂-Triton X-100.

that the maximum emission of three systems (luminol, luminol-H₂O₂ and Triton X-100-luminol-H₂O₂) were also near 425 nm and the Triton X-100-luminol-H₂O₂ system provided the strongest signal. It means that the emitter of the Triton X-100-luminol-H₂O₂ system is also the 3-aminophthalate anion and Triton X-100 could enhance the CL intensity of the luminol-H₂O₂ system. Thus, it was supposed that the function of Triton X-100 was that it could be found in micelles, and the luminol molecule permeated into the micelle so that it could be concentrated in the micelle, and then the quantum yield of the CL reaction was improved and the CL intensity was improved. Critical micelle concentration (CMC) of Triton X-100 is $0.2 \times 10^{-3} \text{ mol} \cdot \text{L}^{-1}$ at 25 °C. In this study, the concentration range of Triton X-100 was between $2 \times 10^{-4} \text{ g} \cdot \text{mL}^{-1}$ and $4 \times 10^{-2} \text{ g} \cdot \text{mL}^{-1}$ (w/v), converting to $0.3 \times 10^{-3} \text{ mol} \cdot \text{L}^{-1}$ – $60 \times 10^{-3} \text{ mol} \cdot \text{L}^{-1}$. It showed that the Triton X-100 concentration was above CMC and supported the viewpoint that the function of Triton X-100 was that it could be found in micelles. In order to confirm the improving effect of the luminol in the Triton X-100 micelle microenvironment, further experimental data was provided in Table 2. From Table 2, it can be seen that Triton X-100 provided obvious enhancing effects on the luminol-H₂O₂ system. The higher the Triton X-100 concentration, the more obvious the enhancing effect of Triton X-100.

Based on the discussions above and combining this with the kinetic curve, the possible mechanism of the present reaction was proposed as follows: (1) Luminol was oxidized by hydrogen peroxide to produce excited state 3-aminophthalate anion; (2) There were micelles with the addition of Triton X-100, and the reaction environment

was improved, luminol molecules permeated into and concentrated in micelles, the reaction was sensitized and the quantum yield of the CL reaction was improved and the CL intensity was enhanced; (3) The excited state 3-aminophthalate anion rebounded to the ground state, producing emission [28,29].

Conclusion

From the above results, it was concluded that this method provides a simple technique for quantitative analysis of Triton X-100. The CL intensity was linear with Triton X-100 concentration in the range of $2 \times 10^{-4} \text{ g} \cdot \text{mL}^{-1}$ – $4 \times 10^{-2} \text{ g} \cdot \text{mL}^{-1}$ (w/v). The detection limit was $3.97 \times 10^{-5} \text{ g} \cdot \text{mL}^{-1}$ (w/v). The desirable recovery ratio ensured the accurate detection of Triton X-100 in environmental water samples. The mechanism was also investigated in detail by fluorescence spectrum and kinetic curve analysis.

Experimental

Reagents

All chemicals were of analytical reagent grade and doubled distilled water was used throughout. Luminol and Triton X-100 were purchased from Sigma Corporation. The $0.01 \text{ mol} \cdot \text{L}^{-1}$ luminol stock solution was prepared by dissolving 0.1772 g luminol with 5 mL $1 \text{ mol} \cdot \text{L}^{-1}$ NaOH solution and doubled distilled water to 100 mL. The luminol solution was stable for at least 1 week when stored in the refrigerator at 4°C . Working standard solutions of luminol were freshly prepared from the stock solution by appropriate dilutions with $0.2 \text{ mol} \cdot \text{L}^{-1}$ Na_2CO_3 before use and pH adjusted to 12.5 with $1 \text{ mol} \cdot \text{L}^{-1}$ NaOH. A standard solution of Triton X-100 (8% w/v) was prepared by dissolving 8 g Triton X-100 with doubly distilled water to 100 mL. Working standard solutions of Triton X-100 were freshly prepared from the stock solution by appropriate dilutions.

Apparatus

The CL signal was measured with a static method by a BPCL ultra-weak luminescence analyzer (Institute of Biophysics, Chinese Academy of Science, Beijing, China). The CL intensity, amplified by a sensitive photomultiplier tube (PMT) operated at -400 V, was measured under the control of a computer. Fluorescence spectra were obtained with a RF-5301PC fluorescence spectrophotometer (Shimadzu, Japan).

Analytical procedure

The reagent solutions were injected rapidly into the sample pool (each solution was 100 μL) and in turn the reaction door closed instantly so that the reaction was in the dark. At the same time, the computer program was started immediately to record the CL signal. The CL intensity ΔI was calculated by $\Delta I = I_s - I_0$, where I_s and I_0 are the CL signals in the presence and absence of Triton X-100, respec-

tively. All analysis was performed according to this procedure.

Kinetic curve method

100 μL Triton X-100 ($1 \times 10^{-2} \text{ g} \cdot \text{mL}^{-1}$), 100 μL H_2O_2 ($0.4 \text{ mol} \cdot \text{L}^{-1}$) and 100 μL luminol ($1.0 \times 10^{-4} \text{ mol} \cdot \text{L}^{-1}$) were injected into the sample pool in the correct order. According to the analytical procedure, the CL signal was collected and recorded consecutively within 100 seconds. And then the curve of CL intensity-time (kinetic curve) was drawn up.

Sample preparation

Water samples were picked from Nanhu Lake and Tangxun Lake near our campus and also near the countryside. Prior to analysis, the collected water was filtered using a standard $0.45 \mu\text{m}$ cellulose filter. In order to perform the recovery test, a known amount of Triton X-100 standard solution was added to the samples and then the mixture was diluted to suitable concentration with doubled distilled water for analysis.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

All authors contributed to the experimental design. Xiaoyu Liu focused on the experiment design and contributed to drafting the manuscript. Aifang Li and Baohui Zhou carried out nearly all of the laboratory work. Chaokun Qiu, Hongmin Ren deal with the data analysis. All authors read and approved the final manuscript.

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