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Disturbance of potassium iodide solution on nonlinear chemical oscillation system of $(NH_4)_4Ce(SO_4)_4$-$NaBrO_3$-malonic acid

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Abstract

The objective of this paper was to study the effect of potassium iodide solution on nonlinear chemical oscillation system of $(NH_4)_4Ce(SO_4)_4$-$NaBrO_3$-malonic acid in the food safety detection. Taking milk powder and pure water blank samples as tested samples, click the menu to collect data. When oscillation curve went on smoothly to 8 min, potassium iodide solution of different concentration was injected, respectively, and the influence of potassium iodide solution of each concentration on intuitive information and characteristic parameters of the corresponding oscillation spectrum was investigated. The results showed that when the concentration of potassium iodide was higher than 1×10^{-3} mol/L, visual information and some characteristic parameters of nonlinear chemical oscillation spectra of milk powder and pure water blank samples were greatly disturbed, and there was a good linear relationship between oscillatory end time and the concentration of potassium iodide, and correlation coefficient was 0.9902. At the same time, this study provided a reference for the study of disturbance of other components on nonlinear chemical oscillation system in the food safety detection.

Keywords: potassium iodide; chemical oscillation; disturbance; milk powder.

Practical Application: Research about disturbance of potassium iodide solution on nonlinear chemical oscillation system, which provided reference for disturbance of other components on oscillation system in the food safety detection.

1 Introduction

As a typical non-equilibrium and nonlinear phenomenon, chemical oscillation is a kind of disordered homogeneous state. In recent years, nonlinear chemical oscillation theory has been widely studied and applied in various fields. Nonlinear phenomena in natural science can be shown directly by chemical oscillating reaction. Therefore, the research and application of this theory have been developed rapidly. Nonlinear chemical oscillation system is a kind of dynamic fingerprint based on the theory of non-equilibrium chemistry. Characteristic curve of reaction in the chromatogram can provide rich chemical information, which has a broad application prospect in analytical chemistry. Nonlinear chemical oscillation system is used in analysis and detection, which is mainly based on the relationship between the quantity of tested substance and quantifiable information of nonlinear chemical oscillation spectrum (such as induction time, oscillation period, maximum amplitude, oscillatory end time, etc.). When tested substance is added, some characteristic parameters of fingerprint can be changed regularly, and then chemical oscillation system is used for quantitative analysis and detection of tested substances. At present, many research results had been reported on the reaction mechanism (Field & Schneider, 1989; Taylor, 2002) and application (Gan et al., 2002; Wang et al., 2005; Gao et al., 2003) of nonlinear chemical oscillation system (Ma et al., 2016; Fang et al., 2010; Ma, et al., 2017). However, there were few reports on the disturbance of nonlinear chemical oscillation system, especially in the application of dairy quality control.

Milk and dairy products are rich in nutrients, including protein, lipids, sugars, minerals (phosphorus, potassium, calcium, magnesium, iron) and trace elements. They are important sources of human nutrition. So far, the consumption of dairy products is increasing year by year, especially infant formula milk powder. It is the most important source of nutrition for non-breastfeeding infants (Ponnal et al., 2021), and has become a product with a rapid growth rate in Chinese dairy consumption. Potassium iodide is a kind of colorless and transparent granular powder, and it is easy to deliquesce in wet air and soluble in water. Potassium iodide is an iodine source in organic synthesis. In addition, potassium iodide can also be used as a catalyst in nucleophilic substitution reaction, so as to enhance the activity of chlorinated hydrocarbon, brominated hydrocarbon or methanesulfonate as nucleophilic substituents (Kanagan & Pari, 2019; Huang & Bartell, 2001; Raju et al., 2019). In oscillating system, because the concentration of components, reaction rate and other variables can change with time, the shape of oscillation curve can be changed if the reaction of a certain element is affected when interfering substance is added into the system. This can provide a lot of intuitive and quantitative information, and can be used as the basis for application in analysis and detection of the food safety.

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In this paper, taking infant formula milk powder as sample, nonlinear chemical oscillation system of $(NH_4)_4Ce(SO_4)_4$-$NaBrO_3$-malonic acid was used for sample analysis. Different concentrations of potassium iodide solution interfered with the system, which changed the concentration of substrate. At the same time, other chemicals coexisting with the substrate in the sample had the ability to inhibit and interfere with oscillation reaction. Characteristic parameters of spectrum, such as induction time, oscillatory end time and oscillation life, were changed obviously. According to the change of characteristic parameters of the corresponding chromatogram, it was found that the disturbance of nonlinear chemical oscillation system of $(NH_4)_4Ce(SO_4)_4$-$NaBrO_3$-malonic acid could be identified by intuitive information of fingerprint (such as induction curve, wave curve, peak shape, etc.) and quantifiable characteristic parameter information when the concentration of potassium iodide was more than 5×10^4 mol/L. This study provided a theoretical basis for the study of the disturbance of other components in the food to nonlinear chemical oscillation system in the food safety detection.

2 Materials and methods

2.1 Reagents and materials

The solutions of 1.00 mol/L sulfuric acid, 1.00 mol/L malonic acid, 0.01 mol/L sodium bromide, 0.80 mol/L sodium bromate, 0.05 mol/L cerium ammonium sulfate and potassium iodide used in the experiment were analytically pure and purchased from Tianjin Fuchen chemical reagent factory. Double distilled water was used throughout. Infant formula milk powder, hereinafter referred to as "milk powder", was purchased in shengdemei supermarket.

2.2 Instrumentation

A nonlinear chemical fingerprint instrument (Model MZ-1B-2) developed by Central South University and Xiangtan Ltd. (Hunan, China) was used. The electrodes were 217 calomel and 213 platinum (Shanghai Precision & Scientific Instrument Co., China). 217 calomel electrode was used as reference electrode, and 213 platinum electrode was used as working electrode. Electronic balance (Model BS 224 S) was developed by Shanghai Precision & Scientific Instrument Co., China.

2.3 Fingerprint determination of nonlinear chemical oscillation system

Take 0.70 g milk powder sample into the reactor, and add 1.00 mol/L malonic acid solution 10.00 mL, 1.00 mol/L sulfuric acid solution 25.00 mL, 0.01 mol/L sodium bromide solution 1.00 mL, 0.05 mol/L cerium sulfate solution 3.00 mL and 15.00 mL distilled water, respectively. Cover the reactor with injection hole and electrode. Open the constant temperature system to adjust the reactor, set the temperature at 50 °C, and set the constant speed stirring at 800 r/min. Click the menu to collect data. When stirring to 3.00 min, quickly add 0.80 mol/L sodium bromate solution 5.00 mL with syringe. E-T curve was recorded until potential did not change with time. Quantitative parameters such as induction time, maximum amplitude and oscillation period were processed by MATLAB (ver. 7.11.2010b).

2.4 Preparation of potassium iodide solution

Potassium iodide solutions of 1 mol/L, 8×10^{-1} mol/L, 1×10^{-1} mol/L, 9×10^{-2} mol/L, 7×10^{-2} mol/L, 5×10^{-2} mol/L, 3×10^{-3} mol/L, 1×10^{-3} mol/L and 5×10^{-4} mol/L were prepared by GB 601-77. 3ml of the above reagents were used to interfere with nonlinear chemical oscillation system of milk powder and blank samples to investigate the influence of different concentrations of potassium iodide solution on the corresponding oscillation patterns.

3 Results and discussion

3.1 Experimental principle

Experimental equipment of nonlinear chemical oscillation system is mainly composed of reaction device, magnetic stirrer, super constant temperature device, data acquisition device, electrode potential signal processing device and computer system. Based on the principle of nonlinear chemical reaction, the instrument uses different chemical components in different samples as dissipative under specific experimental conditions. Data acquisition and conversion and precision constant temperature sensing system are used to continuously detect and record the information in the process of chemical reaction. Thus, oscillation spectrum of tested sample is obtained. The technique is to use different samples because of their different chemical composition and content, which leads to the difference of oscillation spectrum of each sample. Through these differences, the samples can be qualitatively identified and quantitatively analyzed. The device is shown in Figure 1.

3.2 Essential information in nonlinear chemical fingerprint of milk powder

In this study, nonlinear chemical fingerprint was obtained by adding 0.70 g of milk powder into the reactor, and the fingerprint was shown in Figure 2. It was obvious from Figure 2 that essential characteristic information of nonlinear chemical fingerprint mainly included inductive time (t_{ind}), undulatory period (τ_{und}), undulatory life (t_{und}), canyon time (t_{can}), canyon potential (E_{can}), peak top potential (E_{pet}), oscillatory end potential (E_{une}), peak top time (t_{pet}), oscillatory start potential (E_{uns}), oscillatory end time (t_{uns}), maximum amplitude ($\Delta E_{\max}$) and so on. All of them were described in detail in the literature (Ma et al., 2017). Oscillation wave shape of the fingerprint could reflect the characteristics of complex samples, and quantitative information was very important and helpful for distinguishing and evaluating of adulterated milk powder.

3.3 Basic oscillation phenomena of nonlinear chemical oscillation

In the open system, when tested substance and reagent are mixed in the reactor, chemical oscillation curve appears immediately, and the reaction system continuously exchanges

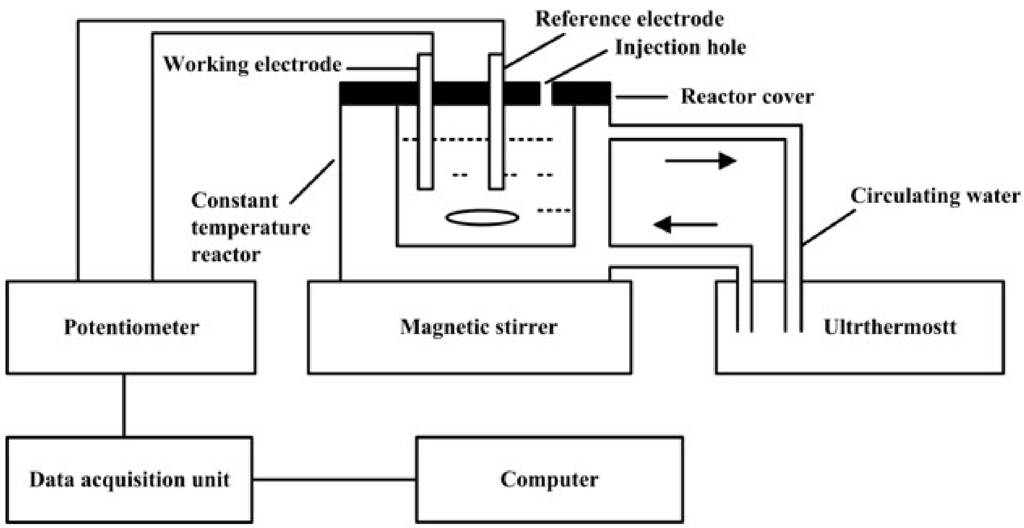

Figure 1. Schematic diagram of nonlinear chemical oscillation system.

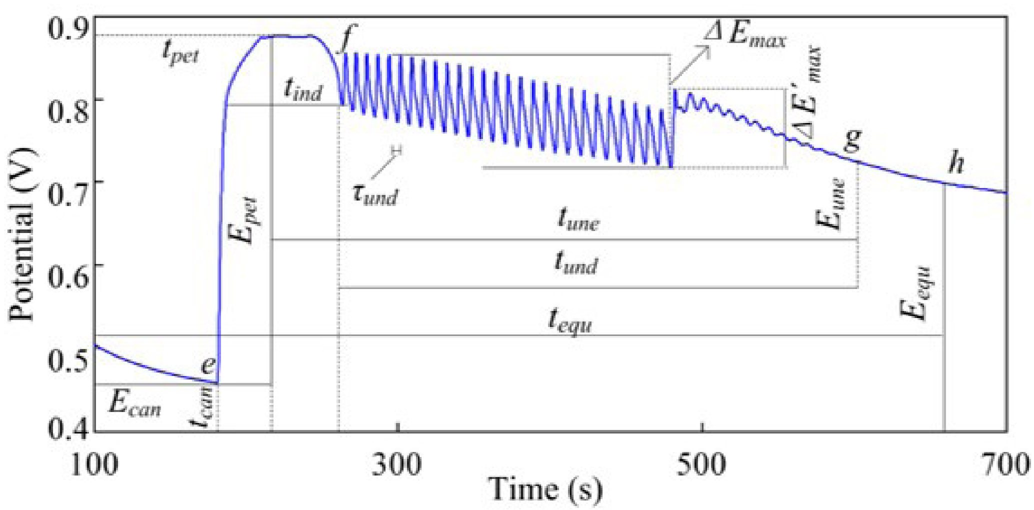

Figure 2. Basic characteristic information of oscillation spectrum of milk powder. e-f: inductive curve; f-g: fluctuation curve; g-h: stop wave curve; point e and h are the start point and end point of reaction, respectively.

materials and energy with the outside world, so that oscillating reaction can go on all the time. Therefore, oscillating reaction can be carried out continuously and stably in the open system, but it has a certain influence on quantitative analysis of samples.

Nonlinear chemical oscillation system in this study was carried out in a closed system. With the passage of time and consumption of reactants, the system could not obtain new substances from the outside, and eventually it would enter equilibrium state and oscillation behavior would stop. When sodium bromate solution was added into milk powder nonlinear chemical oscillation system, interference diagram of potassium iodide solution on the spectrum was shown in Figure 3a. Figure 3b was a partial diagram. Experimental results showed that electric potential was raised rapidly, the solution was changed from colorless to light yellow, at the same time, Br_2 was discharged, and oscillation curve entered induction period. After induction period of about 260 s, the potential decreased and the curve entered oscillation period. Oscillation spectrum and period were relatively stable, and oscillation periodic wave was stable. The trajectory was shown in Figure 3b. In process I, because the reaction was carried out in acidic medium, BrO– 3 participated in the redox reaction with ammonium cerium sulfate as catalyst. The bromine ion was consumed, $HBrO_2$ was accumulated and Br_2 was produced. With the extension of reaction time, the concentration of bromine ion in the system decreased gradually (Ma et al., 2017; Huang & Bartell, 2001). When the concentration of bromine ion was lower than the critical concentration, the whole oscillating reaction was controlled by process II. Figure 3b showed the process from position A to position B of oscillation curve. In this process, $HBrO_2$ was an important intermediate

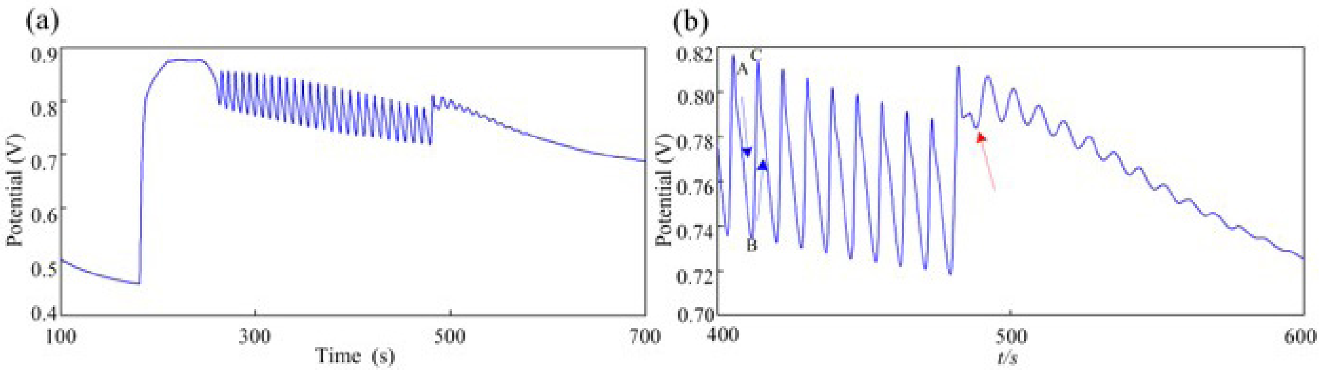

Figure 3. Interference of 1×10^{-1} mol/L potassium iodide solution on oscillation spectrum of milk powder. (a) Integral graphic, (b) Corresponding local graphic.

product, which controlled the conversion of process B to C. Meanwhile, cerium ion changed from Ce^{3+} to Ce^{4+}, and the color of solution changed to colorless from point A to point B. The accumulation of Br2 and HOBr would initiate process III. However, the accumulation of HBrO2 was hindered by the presence of a large number of Br2. The methylene in malonic acid molecule could react with Br2 due to the activation of two carboxyl groups. The product was shown in Equation 7, that was, in acidic medium, malonic acid could remove Br2 from the system, thus regenerating Br–. From process B to process C, the color of solution changed to light yellow. The next new cycle would begin when Br- concentration accumulated again. In this way, process I, process II and process III moved circularly to form oscillating reaction. At the same time, the color of the solution was changed periodically between light yellow and colorless. Bubbles were generated in the whole oscillating reaction process, which indicated that carbon dioxide was generated in the oscillation process.

When oscillation curve was stable, 1×10^{-1} mol/L potassium iodide solution was injected into the system at 8 min. A new oscillation phenomenon was occurred. As shown by the red arrow in Figure 3b, the potential rose abruptly. After oscillation period increased, it suddenly became shorter. Then oscillation period became shorter and shorter, and the amplitude decreased gradually. Finally, oscillation state stopped and reached equilibrium state. The experimental results were shown that the addition of potassium iodide solution hindered the whole oscillating reaction and made the whole oscillating reaction quickly entered into equilibrium state.

Process I (Equations 1-3):

$$Br^- + BrO_3^- + 2H^+ \rightarrow HBrO_2 + HOBr \quad (1)$$

$$HBrO_2 + Br^- + H^+ \rightarrow 2HOBr \quad (2)$$

$$Br^- + HOBr + H^+ \rightarrow H_2O + Br_2 \quad (3)$$

Process II (Equations 4-6):

$$BrO_3^- + H^+ + HBrO_2 \rightarrow 2BrO_2^\bullet + H_2O \quad (4)$$

$$H^+ + Ce^{3+} + BrO_2^\bullet \rightarrow Ce^{4+} + HBrO_2 \quad (5)$$

$$2HBrO_2 \rightarrow HOBr + H^+ + BrO_3^- \quad (6)$$

Process III (Equations 7-9):

$$CH_2(COOH)_2 + Br_2 \rightarrow Br^- + H^+ + BrCH(COOH)_2 \quad (7)$$

$$4Ce^{4+} + BrCH(COOH)_2 + 2H_2O \rightarrow 4Ce^{3+} + HCOOH + Br^- + 5H^+ + 2CO_2 \quad (8)$$

$$HOBr + HCOOH \rightarrow Br^- + H^+ + H_2O + CO_2 \quad (9)$$

3.4 Effect of different concentrations of potassium iodide on milk powder nonlinear chemical oscillation system

The menu was immediately dotted for collecting data after adding 0.70 g milk powder sample into the reactor. When constant stirring rate just lasted for 3.0 min, 5.00 mL of 0.80 mol/L sodium bromate solution was injected into the reactor, and the potential rose rapidly. After a period of induction, the spectrum entered oscillation state. When oscillating reaction proceeded smoothly for a period of time, different concentrations of potassium iodide solution were injected into the reactor at 8 min. With the different concentration of potassium iodide solution injected, the corresponding nonlinear chemical oscillation spectrum of milk powder had certain difference. At this time, the color of the solution was changed between light yellow and colorless. The specific spectrum was shown in Figure 4a. In Figure 4a, sample 1 milk powder spectrum was not added with potassium iodide solution. When mixed reagent and milk powder sample in the reactor were dissipated, oscillation state stopped and the spectrum entered into equilibrium state. In Figure 4a, different concentrations of potassium iodide solution were added to milk powder samples of sample 2 to sample 9. With the increase of potassium iodide solution concentration, characteristic parameters of nonlinear chemical oscillation spectrum of milk powder had obvious changed, such as induction time, oscillatory end time, undulatory life and wave number, as shown in Figure 4b-4c. According to the literature (Zhou et al., 2011), some traditional Chinese medicine and food showed damping effect on the reaction of nonlinear chemical oscillation system. When the amount of tested substance was too small or measured substance

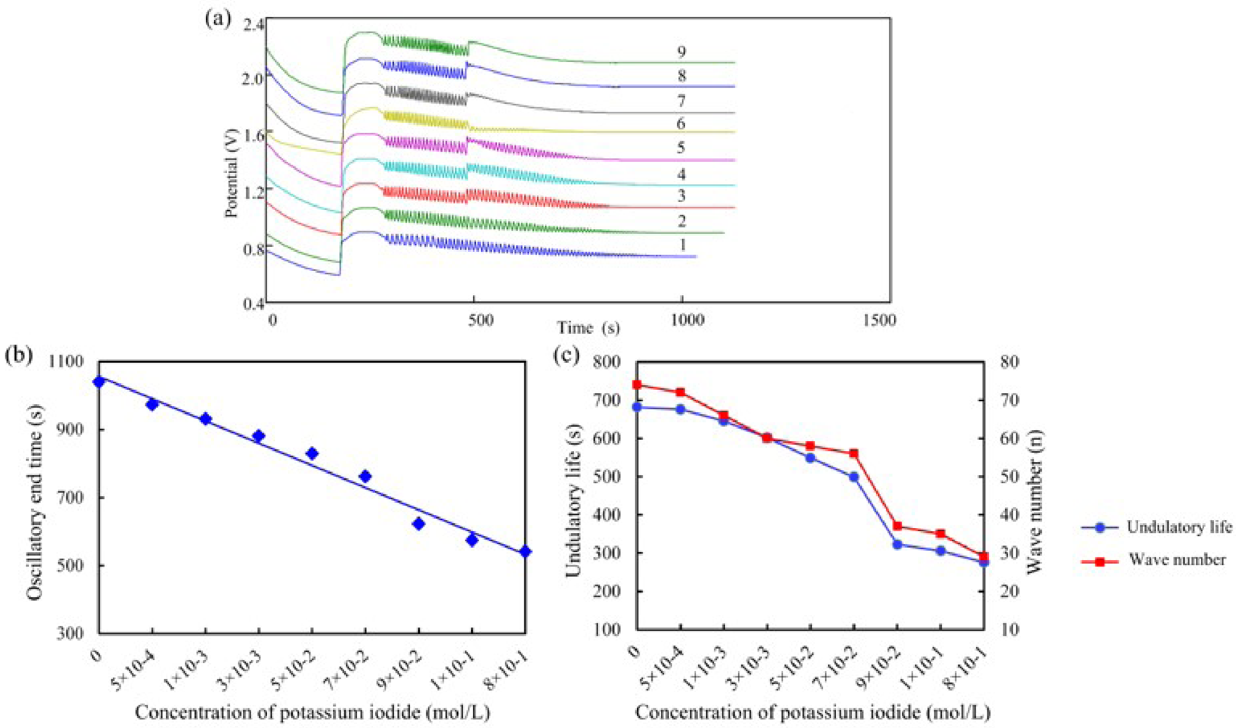

Figure 4. Effect of potassium iodide solution on oscillation spectrum of milk powder: (a) for effect of different concentrations of potassium iodide solution on oscillation spectrum of milk powder, (b) for effect of potassium iodide solution on oscillatory end time of oscillation spectrum of milk powder, (c) for effect of potassium iodide solution on undulatory life and wave number of oscillation spectrum of milk powder. 1-0, 2-5 × 10^{-4} mol/L, 3-1 × 10^{-3} mol/L, 4-3 × 10^{-3} mol/L, 5-5 × 10^{-2} mol/L, 6-7 × 10^{-2} mol/L, 7-9 × 10^{-2} mol/L, 8-1 × 10^{-1} mol/L, 9-8 × 10^{-1} mol/L.

was different, the concentration of the substrate participating in the reaction was very low in the sample, and inhibition effect of the substance coexisting with the substrate was very weak. The time required for inducing oscillation reaction would be prolonged, and oscillation period would also be prolonged (Tan et al., 2018). It could be seen from Figure 4b-4c that with the increase of potassium iodide concentration, the concentration of substrate participating in the reaction was also increasing. Not only oscillatory end time and undulatory life were gradually shortened, but also wave number was reducing, and oscillatory end time was also increasing. At the same time, the ability of other chemicals coexisting with the substrate in the sample to inhibit and interfere with oscillating reaction was also significantly increased, leading to the complete prevention of oscillation reaction (Tan et al., 2018; Gao et al., 2007; Zhang et al., 2012). At this time, the color of solution also was changed accordingly from original light yellow and colorless to the alternation of yellowish brown and colorless. It may be due to the reaction between potassium iodide and bromine formed in the system and the precipitation of iodine, and some iodine is dissolved in water and makes the solution yellow or yellowish brown.

There was a good linear relationship between characteristic parameter oscillatory end time and the concentration of potassium iodide, and correlation coefficient was 0.9902. When the concentration of potassium iodide was 9 × 10^{-2} mol/L, characteristic parameters oscillatory end time, undulatory life and wave number decreased sharply. When the concentration of potassium iodide increased gradually, the characteristic parameters decreased relatively smoothly. When the concentration of potassium iodide was greater than 8 × 10^{-1} mol/L, oscillation state stopped immediately and wave number disappeared. This conclusion was consistent with the change trend of intuitive graph in Figure 4a.

3.5 Effect of potassium iodide solution on nonlinear chemical oscillation system of blank sample

In the closed system, blank system was composed of sulfuric acid, malonic acid, sodium bromated, sodium bromide, cerium ammonium sulfate and water. With the passage of time and material consumption, the system could not obtain new substances from the outside, and finally it would enter equilibrium state, and oscillation behavior would stop. It could be seen from Figure 5a that the whole oscillation curve entered induction period first, oscillation period and oscillation spectrum changed greatly, and the amplitude was irregular, which eventually leaded to the instability of the whole oscillation curve. Due to small amount of dissipative material in the blank sample water, redox reaction could not be finished in a short time, so redox reaction time was prolonged. When 5000 s, dissipative material in the reactor was basically consumed, oscillation period and maximum amplitude gradually decreased and finally disappeared, and oscillation curve reached equilibrium state. The reaction mechanism of the whole oscillation process was described (Section 3.3).

1×10^{-1} mol/L potassium iodide solution was injected into the system at 8 min when oscillation curve was stable, a new oscillation phenomenon occured, as shown by the red arrow in Figure 5b. Compared with Figure 3b, when potassium iodide was injected, the potential did not rise abruptly, but suddenly became shorter after oscillation period increased, and oscillation period began to lengthen and the amplitude increased correspondingly.

3.6 Effect of different concentrations of potassium iodide on nonlinear chemical oscillation system of blank sample

After adding 5.00 mL blank distilled water into the reactor, click the menu to collect data. When constant stirring rate just lasted for 3.0 min, 5.00 mL of 0.80 mol/L sodium bromate solution was injected into the reactor, and the potential rose rapidly. After a period of induction, the spectrum entered into oscillation state. When induction time was about to reach the peak, a small wave valley appeared, as shown by the arrow in Figure 6a. When oscillating reaction proceeded smoothly for a period of time, different concentrations of potassium iodide solution were injected into the reactor at 8 min. With the different concentration of potassium iodide solution injected, corresponding blank sample nonlinear chemical oscillation spectrum had certain difference. At this time, the color of solution was changed between light yellow and colorless, and specific spectrum was shown in Figure 6a. Sample 1 blank sample in Figure 6a showed that no potassium iodide solution was added. When mixed reagent in the reactor was dissipated, oscillation state stopped and the spectrum entered equilibrium state. In Figure 6a, different concentrations of potassium iodide

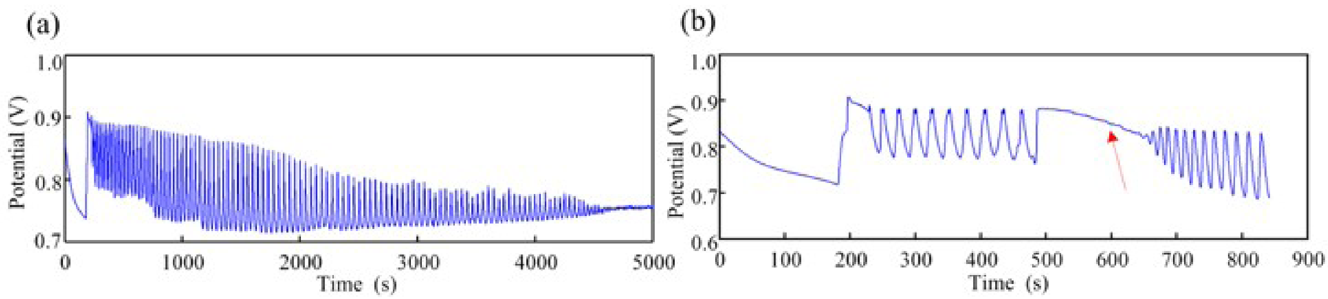

Figure 5. Effect of potassium iodide solution on oscillation system of blank sample: (a) for oscillation spectrum of blank sample, (b) interference of 1×10^{-1} mol/L potassium iodide on blank sample.

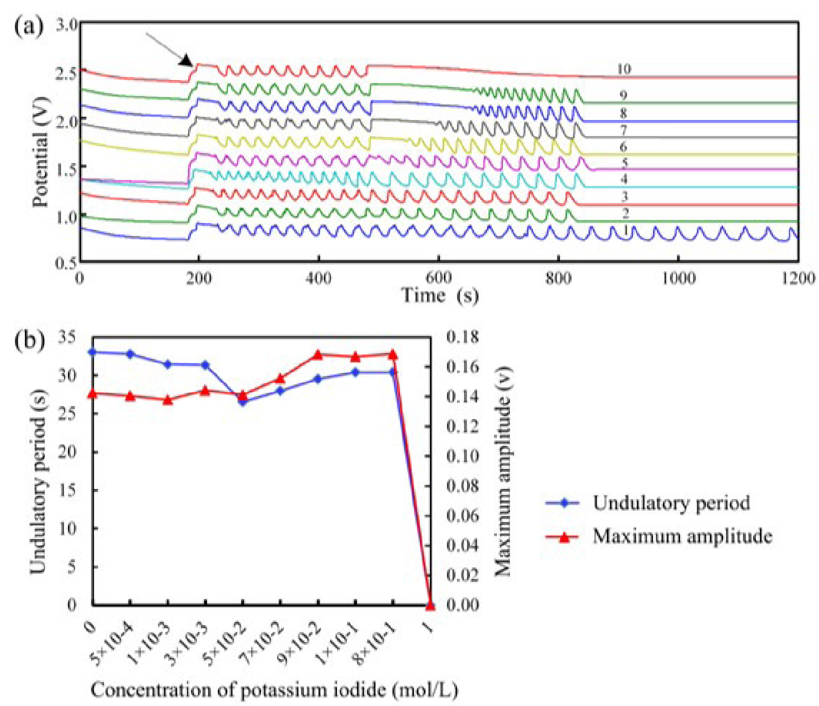

Figure 6. Oscillation spectrum of blank sample: (a) for effect of different concentrations of potassium iodide solution on oscillation spectrum of blank sample, (b) for effect of potassium iodide solution on undulatory period and maximum amplitude of oscillation spectrum of milk powder, 1-0, 2-5 $\times 10^{-4}$ mol/L, 3-1 $\times 10^{-3}$ mol/L, 4-3 $\times 10^{-3}$ mol/L, 5-5 $\times 10^{-2}$ mol/L, 6-7 $\times 10^{-2}$ mol/L, 7-9 $\times 10^{-2}$ mol/L, 8-1 $\times 10^{-1}$ mol/L, 9-8 $\times 10^{-1}$ mol/L, 10-1 mol/L.

solution were added to blank sample 2 to sample 10. With the increase of potassium iodide solution concentration, characteristic parameters of nonlinear chemical oscillation spectrum of blank samples had obvious changes between 480 s and 900 s, as shown in Figure 6a-6b. With the increase of the concentration of potassium iodide, the concentration of the substrate participating in the reaction also increased correspondingly, and the ability of other chemical substances coexisting with the substrate in the sample to inhibit and interfere with oscillating reaction was also significantly increased, as shown in the sample map 10 in Figure 6a-6b, eventually leading to the complete prevention of the occurrence of oscillation reaction (Kanagan & Pari, 2019).

When the concentration of potassium iodide was less than 1×10^{-3} mol/L, the influence of potassium iodide solution on characteristic parameters of oscillation spectrum of blank sample was not obvious. When the concentration of potassium iodide was greater than 1×10^{-3} mol/L, there was no obvious change rule in induction period, and oscillation period and amplitude were changed greatly. When the concentration of potassium iodide was 5×10^{-2} mol/L, characteristic parameters such as oscillatory end time, undulatory life and wave number increased gradually. When the concentration of potassium iodide was more than 8×10^{-1} mol/L, inhibition effect of other chemicals coexisting with the substrate on oscillation reaction reached the maximum, which led to immediate stop of oscillation state and wave number basically disappeared. This conclusion was consistent with the change trend of intuitive graph in Figure 6a.

3.7 Interference of potassium iodide on chemical oscillation system and its application in practice

There are many brands and types of milk powder, and the potassium content of different milk powder is different. Milk powder paid attention to balanced nutrition and good digestion and absorption, not the higher the potassium content, the better. High potassium content in milk powder would increase the burden on the baby's intestines, stomach and kidneys and affect digestion. In addition, the inability to absorb potassium could also lead to constipation. Therefore, in order to ensure people's food safety and health, the potassium content in milk powder should be controlled within the standard range. In the actual production process of milk powder, after the milk powder with excessive or insufficient potassium content was added into the reaction tank, the maximum amplitude, fluctuation life and wave stop time after the interference of the characteristic parameters of the corresponding milk powder would change. Therefore, through the changes of characteristic parameters and intuitive map, in the process of milk powder production, when the potassium content exceeded or was insufficient, the interference of potassium iodide on the chemical oscillation system could be identified, so as to ensure the quality of milk powder in the process of online production.

4 Conclusion

In this paper, different concentrations of potassium iodide solution were used to interfere with nonlinear chemical oscillation system of milk powder and pure water blank samples. The experimental results showed that the reaction process and mechanism was basically the same, but for different samples, due to the difference of substrate concentration involved in the reaction, when different concentrations of potassium iodide were injected to interfere with the system, oscillation patterns of milk powder and blank sample changed obviously. When the concentration of potassium iodide was less than 1×10^{-3} mol/L, visual spectrum and characteristic parameters of milk powder and pure water blank samples changed little. When the concentration of potassium iodide was more than 1×10^{-3} mol/L, visual spectrum and characteristic parameters changed significantly. Especially, when the concentration of potassium iodide was greater than 9×10^{-2} mol/L, characteristic parameters of oscillation spectrum of milk powder had a sharp decline in oscillatory end time, undulatory life and wave number. When the concentration of potassium iodide was higher than 8×10^{-1} mol/L, oscillation state stoped immediately and wave number disappeared. There was a good linear relationship between characteristic parameter oscillatory end time and the concentration of potassium iodide, which provided a theoretical basis for quantitative analysis of potassium content in milk powder. Through the experiment, it was found that the existence of iodine ion and potassium ion had certain interference to nonlinear chemical oscillation spectrum of the sample. When the sample was analyzed quantitatively, the existence of iodine ion and potassium ion should be avoided in order to improve the safety of food and reduce the hazard to health.

Conflict of interest

The authors declare that they have no conflicts of interest.

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