7. Cryogel based sensor for sodium hydrosulfite

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Submission date: 02-Feb-2023 03:08PM (UTC+0700)
Submission ID: 2004678308
File name: ._Cryogel_based_sensor_for_sodium_hydrosulfite_determination.pdf (515.67K)
Word count: 1997
Character count: 11032

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Cite as: AIP Conference Proceedings **2094**, 020025 (2019); https://doi.org/10.1063/1.5097494
Published Online: 17 April 2019

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Cryogel Based Sensor for Sodium Hydrosulfite Determination

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Abstract. A sensor for determination of sodium hydrosulfite using a cryogel entrapped 5,5-dithiobis-2-nitrobenzoic acid (DTNB) was developed. This sensor is based on the chemical reaction between sodium hydrosulfite and DTNB reagent yielding a yellow product of 5-mercapto-2-nitrobenzoate which can be easily observed by naked-eyes and digital image analysis. The intensity of the color increased with the sodium hydrosulfite concentration which can be quantified by ImageJ software. The developed test kit provided the linear ranges from 10 to 30 ppm ($R^2 = 0.9839$) with a detection limit of 3.51 ppm and limit of quantification of 11.69 ppm.

INTRODUCTION

Sulfite is one of the oldest and most ubiquitous food additives, which has been widely used as blanching and preservative agents in a large variety of foodstuffs to improve the appearance of foods and prevent bacterial growth [1]. It has the ability to inhibit the color change of food not brown when heated been cut for a long time for this reason, it is used in food products such as dried fruits, conned fruit, canned vegetable, preserve fruit, compote, sprouts, flour products and seafood products.

However, harmful chemicals used in food are allowed to be used in food the prescribed- amount sulfite is a bleach that Thailand permits in the food industry. have determine substance that can be used additives in food according to the Ministry of Public Health include sulfur dioxide, sodium-potassium sulfite, sodium potassium metabisulfite. Normally, if the substance found in low amount the human body, there is an enzyme that can convert sulfite to sulfate, which is not toxic to the body and is excreted from the body by urine. However, exposure of this substance in large quantities can cause harm to the consumer since the toxic effects are different in each person especially in people with asthma, it can cause unconsciousness and death [2]. Food and Drug Administration (FDA) has required to show the label of food contains sulfites more than 10 μg/ml [3].

The bleaching agent not allowed as food additive is sodium hydrosulfite. This substance is generally used in non-food industries such as fishing net, ring net, paper, leather, silk fibers. However, some food fraud practice has found in food to make brighter and more delicious. The most contaminated food samples are sprouts, ginger sliced, pickled bamboo, coconut sugar, preserved durian and meat products. The danger of this type of bleaching if exposed, it will

1st International Conference on Material Science and Engineering for Sustainable Rural Development
AIP Conf. Proc. 2094, 020025-1–020025-5; https://doi.org/10.1063/1.5097494
Published by AIP Publishing. 978-0-7354-1824-0/$30.00

cause dermatitis and it will cause inflammation in the exposed area such as the mouth, throat and stomach. In addition, if c[2]ses chest pain, shortness of breath, stomachache, headache, vomit, diarrhea [4].

Nowadays, many countries have regulation on the residual amount of sulfite in different types of food. Thus, sensitive and reliable methods for m[2]surement of sulfite in foods at very low-level are necessary. Distillation of the samples under acidic condition[2]nd photometry and ion chromatography are the traditional and official methods for sulfite determination in foods. These methods proved to be more sensitive and selective, but most of them are either time-consuming or require rather complicated instruments [5]. Another widely developed method for sulfite determination is biosensor. The biosensor uses biological sensing element and a transducer to determine the analyte in samples. The advantages of sensors and biosensors were high sensitivity, high selectivity, fast analysis and relatively low cost [6]. Sensor and biosensors has been developed in various analytes determination such as glucose [7], cancer marker [8], microalbumin [9] and also some food additive such as sulfite [10]. Furthermore, colorimetric detection of sulfite was also developed using nanoparticle [11].

This study reported a simple method to measure sulfite bleach in a test kit. One of the interesting and popular techniques is colorimetric, using color sensor to measure the change of color. The colorimetric sensor are easy and quick method to measure, no complicated tools needed, low cost analysis, can be applied widely. The fabricated sensor used reagent for measuring sulfite bleach was 5,5-dithiobis-2-nitrobenzoic acid (DTNB). This sensor is based on the chemical reaction between sodium hydrosulfite DTNB reagent yielding a yellow product of 5-mercapto-2-nitrobensoat which can be easily observed by naked-eyes [12].

MATERIALS AND METHODS

Materials

Sodium hydrosulfite,5,5'- dithiobis-2-nitro benzoic acid (DTNB) (Sigma-Aldrich chemie GmbH, Germany), Poly vinyl alcohol (PVA) (ALDRICH, Sigma-Aldrich chemie GmbH, Germany), Hydrochloric acid (HCL) (Loba chemie, Mumbai, India), Glutaraldehyde (Fluka, Sigma-Aldrich chemie GmbH, USA), Disodium dihydrogen phosphate (Lobachemie, Mumbai, India), Sodium dihydrogen phosphate (Lobachemie, Mumbai, India) and Ultrapure water (ELGA Buckinghamshire, England).

Apparatus and measurements

Microplate 96 well, magnetic stirrer, micropipette, analytical balance, pH meter, vortex, freezer, centrifuge tube and laboratories glassware.

Cryogel preparation

The cryogel was prepare using polyvinyl alcohol (PVA) solution of 3.3% (w/v) in ultrapure water at 90 ^{o}C. The pH of the PVA solution was then adjusted to 1.0 with the addition of 5M HCl. The glutaraldehyde solution of 0.5% (v/v) was then added as crosslinking agent, and the mixture was the pour on the microplate 96 well. The polymerization was performed at at -20 ^{0}C for 12 h to allow cryogelation process. The PVA cryogel was then rinse with pure water until the neutral pH achieved. The PVA cryogel was then dip in the 5,5-dithiobis-2-nitrobenzoic acid (DTNB) solution (0.25%, b/v) for 30 minutes.

Sodium hydrosulfite determination

The PVA-DTNB cryogel was put into microcentrifuge tube, added a series concentration of standard sodium hydrosulfite solution (0.5-30 ppm). Allow the reaction of DTNB and sulfite to produce yellow color easily observed visually. The series microcentrifuge tubes with gradient yellow color according to sulfite concentration were then capture the image using smartphone. The resulted digital image was the analyze to make the calibration curve between sulfite concentration and color intensity change.

Linearity, limit of detection and limit of quantification study

Linearity of the analytical method is defined as the ability of the method to obtain test results proportional to the concentration of the analyte (within a given range). Linearity was determined from the plot between the color change intensity (y) and sodium hydrosulfite concentration (x) in the range that a correlation coefficient (r) was greater than or equal to 0.99. The limit of detection (LOD) and limit of quantification (LOQ) were the calculated from the resulted linear equation.

RESULTS AND DISCUSSION

Sodium Hydrosulfite Detection

The standard solution of sodium hydrosulfite in centrifuge tube needs 7 minutes to complete the color change from colorless to yellow (Figure 1). The yellow color formation was the sodium hydrosulfite and 5,5-dithiobis-2-nitrobenzoic acid (DTNB) reaction to produce 5-mercapto-2-nitrobenzoate (Figure 2) to analyze the color intensity it was found that the difference in color intensity at different concentration after subtracting the color intensity of the control it was found that the values increased with the concentration of sulfites.

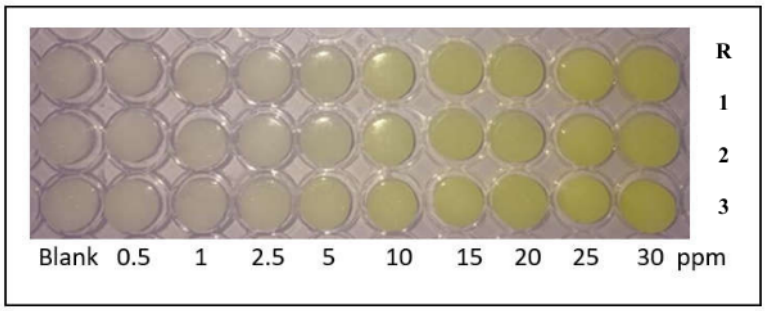

FIGURE 1. The PVA-DTNB color change with the addition of sodium hydrosulfite at various concentration.

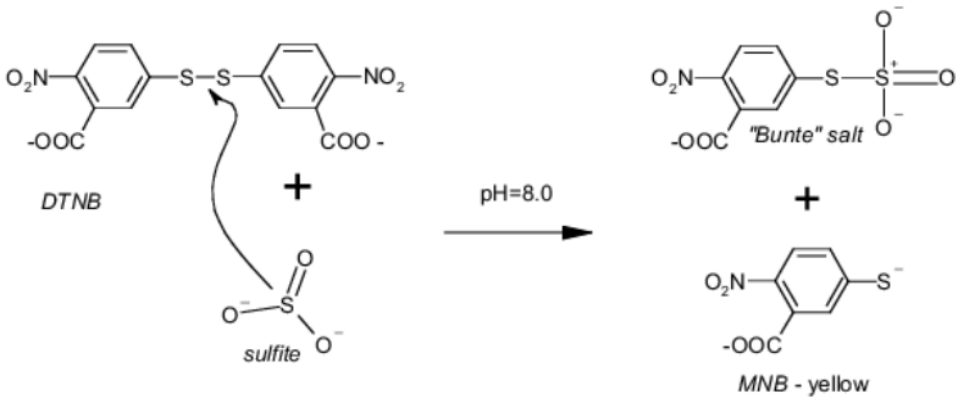

FIGURE 2. Reaction of DTNB in the PVA cryogel with sodium hydrosulfite to produce 5-mercapto-2-nitrobenzoate [13]

Digital image processing

The image was analyzed using the ImageJ software of all channel then bring color intensity create calibration curve relationship between the color intensity of the concentration of sodium hydrosulfite (Figure 3). The blue channel (invert color of yellow) showed the highest sensitivity, thus this blue channel or color intensity was used for further study.

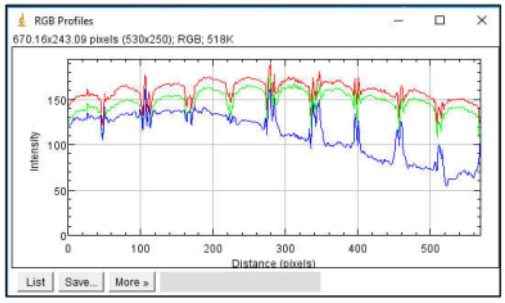

FIGURE 3. Blue channel showed the highest color intensity change of PVA-DTNB cryogel with the addition of sodium hydrosulfite, analyzed by ImageJ software.

Linearity, LOD and LOQ study

Under appropriate conditions, the DTNB reagent concentration of 2.50 mg/L was chosen with a reaction time of 7 minutes. The linear range was obtained from 10 to 30 ppm with a coefficient of determination (R^2) of 0.9839 (Figure 4) the linear regression equation was y = 2.5679x- 14.364. For color visual observation of Sodium hydrosulfite, if was found that the color seen with the naked eye was yellow color related to the sodium hydrosulfite concentration. This result allows the developed sulfite kit widely applied using naked eyes observation. The calculated limit of detection and limit of quantification were 3.51 ppm and 11.69 ppm respectively.

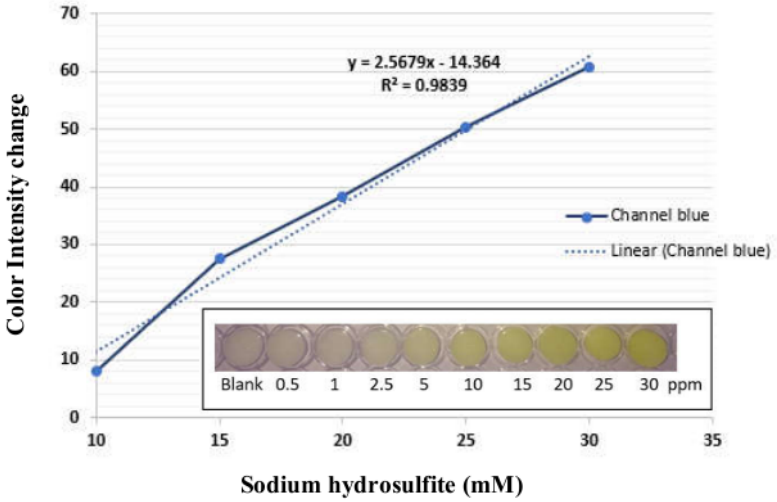

FIGURE 4. The calibration curve between the color intensity and the concentration of Sodium hydrosulfite 10 to 30 ppm. Inset the image of the corresponding PVA-DTNB color change with the sulfite addition.

CONCLUSION

This work reported a colorimetric method for sodium hydrosulfite detection based on the poly vinyl alcohol cryogel as a supporting. The cryogel with porous surface area allow large amount of the reagent of 5,5'- dithiobis-2-nitro benzoic acid (DTNB) which used to react with the analyte of sulfite to produce high intensity color. The fabricated sulfite detection kit showed a good linear range from 10 to 30 ppm (y=2.5679x-14.364, R^2 of 0.9839), with the calculated LOD was 3.51 ppm and LOQ was 11.59 ppm. This poly vinyl alcohol cryogel based sodium hydrosulfite sensor with colorimetric detection would be an excellent model for other colorimetric sensor application and the test kit can be used to detect contaminated of sulfite in the food.

ACKNOWLEDGEMENT

We would like to thank the Biochemistry laboratory of Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Jenderal Soedirman for Student Exchange Program with the Faculty of Science and Technology, Princess of Naradhiwas University, Narathiwat, Thailand.

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