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รายงานสืบเนื่องจากงานประชุมวิชาการระดับชาติด้านวิทยาศาสตร์และเทคโนโลยีเครือข่ายภาคใต้ ครั้งที่ 8 และ งานประชุมวิชาการระดับนานาชาติด้านวิทยาศาสตร์และเทคโนโลยีเครือข่ายภาคใต้ ครั้งที่ 1
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Real-time, Portable and High Selective of Mini-syringe Sensor based on Pararosaniline Hydrochloride Sol-gel for the Formaldehyde Detection in Indoor Air from the Furniture and Poster Shops in Yala Province, Thailand

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Abstract

This research was developed pararosaniline hydrochloride mini-syringe sol-gel formaldehyde sensor by using colorimetric spectroscopy as the transducer. The chemical sensor based on immobilized pararosaniline hydrochloride into sol-gel matrix tetraethyl orthosilicate (TEOS) which is a simple tool that can be used to detect the presence of formaldehyde. Sol-gel sensor was reacted with formaldehyde which can be changed the color from pink to purple, that this sensor used to detect quantitative formaldehyde in the samples. At a wavelength of 539.5 nm, this sensor provided the linear range between 0.75-30 ppm with a detecting limit of 0.26 ppm and sampling time 120 minutes. The high selectivity of sol-gel mini-syringe formaldehyde sensor can be applied to detect formaldehyde from five of advertising and plywood furniture shops (advertising shop of A, B, C, D and plywood furniture shop of E) in Yala Province, Thailand by installing mini-syringe sol-gel sensor inside the shop with three sampling points. From the result, it was found that three advertising shops of A, C and D, which the samples were collected from the vinyl printer area, were detected formaldehyde concentration exceeding the maximum acceptable concentration of the Ministry of Interior's Announcement and higher than the National Institute for Occupational Safety and Health (NIOSH) in the United States. While, both advertising shop B and plywood furniture shop E were not detected. Therefore, mini-syringe sol-gel formaldehyde sensor can be applied to detect toxic formaldehyde gas with the highly selective, easy to use and portable.

Keywords: Formaldehyde, Chemical sensor, Sol-gel, Pararosaniline hydrochloride, Colorimetric transducer

Introduction

Air pollution is contamination of the indoor or outdoor environment by any chemical, physical or biological agent that modifies the natural characteristics of the atmosphere. Especially, formaldehyde (HCHO) is an omnipresent carcinogenic indoor air pollutant (Protano *et al*., 2022). It is the simplest aldehyde, colorless, poisonous gas with a strong smell, that causes severe damage to central nervous system and immune system. Formaldehyde is a major indoor volatile organic compound (VOC) and is classified as a group 1 carcinogen according to the WHO International Agency for Research on Cancer (IARC) (Lemon *et al*., 2007; Salthammer, 2019). Increased exposure of this chemical can increase the risk of cancers in pharynx, nasopharynx, and brain, as well as dermatitis and allergic reactions (Tian *et al*., 2022). Furthermore, it has been reported that formaldehyde is an essential metabolic intermediate formed in all cells during the metabolism of amino acids such as serine, glycine, methionine and choline. However, formaldehyde is also a valuable industrial chemical with limited alternatives. The most common commercially available form is a 30-50% aqueous solution known as formalin. The International Agency for the Research on Cancer (IARC) has identified three main occupational scenarios where workers may be exposed to formaldehyde at air concentrations significantly higher than the indoor and outdoor background levels: (i) the production of formaldehyde and/or its solutions; (ii) the production of products containing formaldehyde or during their use and (iii) the combustion of products generating formaldehyde (Protano *et al*., 2022). Thus, workers in industrial production processes such as resins, plastics, semi-finished wood products, furnishing accessories and textiles (Gonçalves *et al*., 2021), professionals of gross anatomy and pathology laboratories (Tesfaye *et al*., 2021), veterinarians (Menon *et al*., 2021), embalmers (Kishore *et al*., 2021), breeders (Tian *et al*., 2022), carpenters (Rashedi *et al*., 2022), industrial launderers (El-Naggar *et al*., 2022), plywood (Liu et al., 2019), thermal insulating materials (Alade *et al*., 2022), beauticians and printing rooms workers are the categories at higher risk of exposure to formaldehyde (Herrero *et al*., 2022; Siankamari *et al*., 2022). Many of these materials emit low concentrations of formaldehyde over time. Emission levels are highest when materials are new, generally decreasing exponentially, but can take multiple years to reach safe levels. Formaldehyde gas can therefore build up in enclosed areas, particularly when new furnishings or carpeting have been installed and where premises are not adequately ventilated.

Different techniques are available for detection formaldehyde. The spectrophotometric method proposed the reaction between acetylacetone, ammonia and formaldehyde to form 3,5-diacetyl-1,4-dihydrotoludine is the basis of Hantzch reaction and is the principle of spectrophotometric reaction (Almahri & Abdel-Lateef, 2021). But this procedure needs long reaction time and cannot be simply adopted for an automatic analysis. Chromatographic methods such as high performance liquid chromatography (HPLC) and gas chromatography (GC) method having better selectivity, precision and accuracy were for detection of formaldehyde (Dugheri *et al*., 2019; Yuniati *et al*., 2021). These methods all however using long time analysis, need sample preparation step, quite expensive of instrument, complexity and cannot undertake real-time measurement. Especially, HPLC is preferred when formaldehyde is detected in aqueous media. For a semiconductor oxide formaldehyde sensor using SnO_2 nanoflowers were prepared by hydrothermal process that provided a real-time monitoring of low-concentration formaldehyde (Xiang *et al*., 2022). Due to long time of SnO_2 nanoflowers synthesis make it really quite complicated. By contrast, chemical gas sensors could address this with the simple application, reusability, high miniaturization potential and low power consumption at minimal cost, ideal for on-site and distributed monitoring (Cai *et al*., 2022; Han *et al*., 2022; Pan *et al*., 2022). However, electrochemical sensors generally suffer from limited lifetimes and their response can be compromised by cross-sensitivity to other species, including humidity. Therefore, developing simple specific colorimetric formaldehyde sensor is very urgent to give solutions to problems in general public to detect formaldehyde contained in air (Cao & Ma, 2022). For the specific reagent, pararosaniline hydrochloride is interesting reagent used to react with formaldehyde, and the reaction between pararosaniline and formaldehyde will be presented. It involves the absorption of sulfur dioxide in a solution of sodium tetrachloromercurate followed by addition of pararosaniline hydrochloride and formaldehyde to form a purple-colored sulfonic acid derivative of pararosaniline. Moreover, using transparent sol-gels with entrapped sensitive and selective reagents for the detection of formaldehyde, there are many advantages, such as its optical clarity, the ability to entrap specific reagent, thermal and chemical stability, simplicity of preparation and flexibility in controlling its pore size and geometry (Ahmed *et al*., 2022). The key advantages offered by the sol-gel process that is, it can be done at low temperatures and can be modified in a variety of ways. Depending on the substrate, additives and conditions experimental, the synthesized particles or layers of oxide can be controlled to a nanoscale size.

Therefore, this study is to detect formaldehyde via mini-syringe sol-gel sensor using tetraethyl orthosilicate (TEOS) as sol-gel material and shaping as sol-gel granule in mini-syringe with easy and rapid process. The mechanism process, formaldehyde in air system, diffuses sol-gel and reacts with pararosaniline reagent producing the change in observed color of sol-gel that can be detected using colorimetric assay. The specific colors changed sol-gel from pink to violet indicating that air contains formaldehyde. Moreover, the sol-gel mini-syringe formaldehyde sensor can be used to detect formaldehyde contains in air as qualitative and quantitative manner with a cost-effective and uncomplicated method that sufficient levels of sensitivity and selectivity. This developed method is expected to be applied for formaldehyde determination indoor air of the furniture and poster companies in Yala Province, Thailand.

Objectives

This research aim is to present a low-cost, high selectivity and handheld mini-syringe sol-gel formaldehyde sensor with validated performance in indoor air.

Methods

Reagents and Chemicals

All the solvents, reagents and chemicals used to generate this work were analytical grade. Formalin (Formaldehyde solution in aqueous medium, 37%), pararosaniline hydrochloride, tetraethyl orthosilicate, methanol, benzaldehyde, acetaldehyde and toluene were purchased from Merck, Germany. Sodium sulphite and hydrochloric acid were bought from Ajax Chemical, Australia. Ethanol was bought from DaeJung, Korea. Acetone and cetyl trimetylammonium bromide are products of Loba Chemie, Thailand. Analytical grade chemicals were used. Millipore water was used to prepare the experimental solutions. Formaldehyde stock solution was prepared by diluting 37%(w/v) solution for preparation of standard working solution. Stock solution of pararosaniline hydrochloride was prepared by dissolving 0.03 g of pararosaniline hydrochloride, 2.5 mL of hydrochloric acid (2 M), 1.25 mL of sodium sulphate (0.08 M) in 10 mL of deionized water.

Spectrum study of pararosaniline hydrochloride reagent

In a spectrum study of pararosaniline hydrochloride reagents undergoing a specific reaction with formaldehyde. The maximum absorbance wavelength (λ_{max}) was determined by pipetting 9 µL of pararosaniline hydrochloride reagent into cuvette then add 2,991 microliters of deionized water and stir the solution. The absorbance was measured at a wavelength of 350-800 nm by using UV-Visible spectrophotometer was purchased from Jasco International Co., Ltd.

The study of the observed color change between pararosaniline hydrochloride and formaldehyde

The color change of pararosaniline hydrochloride with formaldehyde was studied to determine the selectivity of the reagent versus formaldehyde. Comparing the color produced when pararosaniline hydrochloride reacts with formaldehyde at the concentrations of 0.75, 1.5, 3.0, 4.5, 6.0, 7.5, 15 and 30 ppm.

Evaluation of the reaction between pararosaniline hydrochloride and formaldehyde

For the evaluation of the reaction between the reagent and formaldehyde, the optimum conditions such as sensitivity, linear range and limit of detection were studied. By pipetting 450 µL of pararosaniline hydrochloride solution into the cuvette. Formaldehyde solution was then added to obtain a final concentration of 0.75, 1.5, 3.0, 4.5, 6.0, 7.5, 15 and 30 ppm, respectively. Add deionized water and stir the solution then the absorbance was measured at wavelength 539.5 nm (maximum absorbance wavelength).

Studies on the sampling time of the reaction between pararosaniline hydrochloride and formaldehyde

Duration affects the reaction, therefore, the reaction time between pararosaniline hydrochloride and formaldehyde solution was studied at 60, 120, 180, 240, 300, 360 and 420 minutes. By pipette 450 µL of pararosaniline hydrochloride solution in disposable cuvette, formaldehyde solution was then added to obtain a final concentration. The absorbance was measured versus reaction times at 539.5 nm.

Sol-gel solution preparation

Tetraethyl orthosilicate (TEOS) was used as a gelling agent and cetyltrimethyl ammonium bromide was used as a strong agent for reducing the surface tension that pararosaniline hydrochloride and tetraethyl orthosilicate can be dissolved by simply adjusting. In this work, sol-gel solution was prepared by mixing 1500 μL of pararosaniline hydrochloride solution with 500 μL of hydrochloric acid, 250 μL of sodium sulfide solution, 2,000 μL of ethanol, 1750 μL of deionized water and tetraethyl orthosilicate 4,500 μL. The mixture was then magnetically stirred for 5 h and then 5 drops of cetyltrimethyl ammonium bromide solution was placed in the mixture and stirred for 30 minutes at room temperature. This sol-gel solution was used as the stock sol-gel solution throughout.

Preparation of mini-syringe sol-gel formaldehyde sensor

In the present work, mini-syringe sol-gel sensor was prepared by mixing the sol–gel solution and pararosaniline hydrochloride solution in a 1:1 ratio. 300 μL of the formaldehyde solution was pipetted into mini-syringe or disposable cuvette with care being taken that the solution did not adhere at the walls which would otherwise lead to variable responses. The sol–gel mixture was left at room temperature for 30 min to give a horos color gel containing entrapped pararosaniline hydrochloride. The sol–gel was stored in the freezer to avoid the evaporation of any components. A reference sol–gel was prepared in the same time and stored under the same conditions for use as a blank sample for absorbance determination.

Study on the selectivity of the sol-gel formaldehyde sensor

In order to verify the selectivity of the sol-gel formaldehyde sensor, we selected five compounds with similar and different structure by using methanol (CH_3OH), acetaldehyde (C_2H_4O), benzaldehyde (C_7H_6O), toluene (C_7H_8) and acetone (C_3H_6O) as negative control. n the detection process, the formaldehyde concentration was 30 ppm and the concentration of each kind of other negative controls in the detection system was 300 ppm, which was 10 times higher than target solution. By setting sol-gel in a closed system with 120 min (optimal reaction time) for the vapors react with pararosaniline hydrochloride in sol-gel.

Results

Spectrum of pararosaniline hydrochloride reagent

The first step in parameter optimization is to finding the optimal wavelength, based on the scanning the spectrum of blank solution and pararosaniline hydrochloride solution. From the spectrum of the reagent was found that the maximum absorbance wavelength (λ_{max}) was 539.5 nm as presenting in Figure 1, which is the analytical quality data for this reagent.

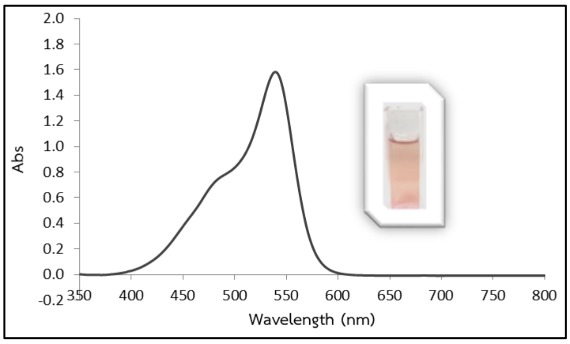

Figure 1 Spectrum profile of pararosaniline hydrochloride reagent.
(Inset: Pararosaniline hydrochloride reagent solution in a cuvette)

The visible color change between pararosaniline hydrochloride and formalin

The order of addition is changed that the formaldehyde and acid-bleached pararosaniline are mixed first, and the sulfide solution then added, color developed occurs. The acidity of the resultant solution prevents the formaldehyde and sulfite from reacting and, presumably, the formaldehyde and pararosaniline react instead. This type of reaction is acid-catalyzed and the product is likely to be stabilized by conjugation of the imine with the phenyl ring. The Schiff base may combine with sulfur dioxide under acid conditions to form an alkylsulfonic acid (Gani *et al*., 2013) is shown in Figure 2.

In the experimental, the color change was observed with the naked eye of pararosaniline hydrochloride reacts with formaldehyde at the concentrations of 0.025, 0.050, 0.10, 0.15, 0.20, 0.25, 0.50 and 1.0 mM in the aqueous phase. From the result found that a strong purple was obtained when high concentration of formaldehyde reacted with pararosaniline hydrochloride, whereas the product of reaction provides a weaker purple at the low concentration according to Figure 3.

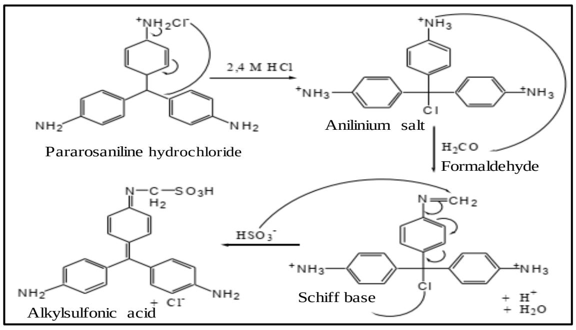

Figure 2 The reaction of formaldehyde with pararosaniline hydrochloride.

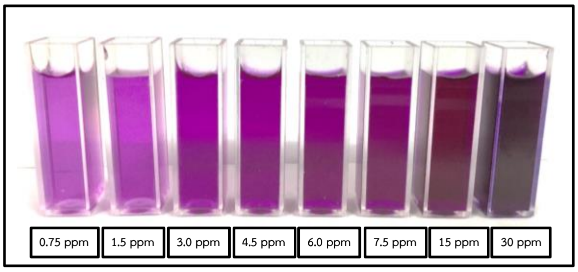

Figure 3 The color of the reaction between pararosaniline hydrochloride and different concentrations of formaldehyde.

Evaluation of the interaction between pararosaniline hydrochloride and formaldehyde

The reaction of pararosaniline hydrochloride and formaldehyde with formaldehyde were studied by adding eight different concentrations of formaldehyde into of pararosaniline hydrochloride solution to obtain 0.75, 1.5, 3.0, 4.5, 6.0, 7.5, 15 and 30 ppm formaldehyde concentrations. The spectrum demonstrated the maximum adsorptions observed for the product was found to occur at wavelength of 539.5 nm. The purple was obtained when formaldehyde reacted with pararosaniline hydrochloride. Due to the faster reaction and stronger color with pararosaniline hydrochloride, this method was chosen for the development of the visual approach for formaldehyde detection. From the reaction between pararosaniline hydrochloride solution and formaldehyde, was found to provide a linear response ranging from 0.75 to 30 ppm and the lowest measurement limit of 0.25 ppm in Figure 4.

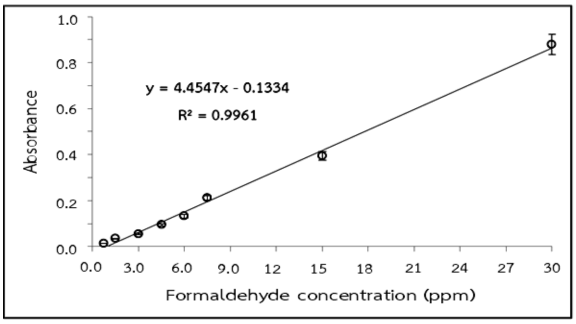

Figure 4 The relationship between absorbance and formaldehyde concentration.

Sol-gel formaldehyde sensor

The sol–gel solution and pararosaniline hydrochloride solution with 1:1 (v/v) ratio was placed in a 1.5 mL disposable cuvette and allowed to set a clear sol-gel then nitrogen was passed though the mixed solution for 1 min to remove any bubbles that can form in the sol-gel.

Figure 5 shows the absorbance after exposing sol-gel sensors to formaldehyde vapor, that provided the linear response ranges from 0.75 to 30 ppm (R^2 = 0.9992) with a minimum measurement limit of 0.25 ppm.

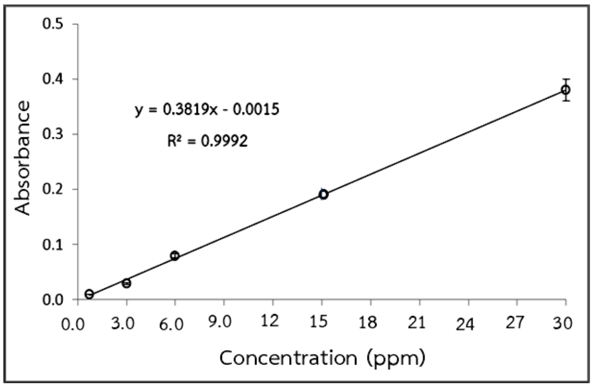

Figure 5 Response of sol-gel formaldehyde sensing exposed to formaldehyde vapor.

Sampling time of sol-gel formaldehyde sensor

From the reaction between pararosaniline hydrochloride in the sol-gel and formaldehyde was found that at various sampling times (60, 120, 180, 240, 300, 360 and 420 min) provided the different sensitivities. This result indicated that the sampling time of 120 min gave the highest analytical sensitivity that was not affected by the evaporation of the sol-gel components, in Figure 6. That is the sampling time when the reaction is most complete and takes the least time.

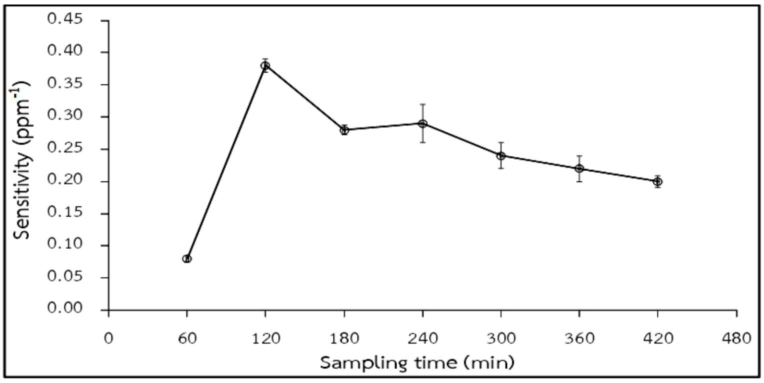

Figure 6 The relationship between analytical sensitivity and sampling time.

Selectivity of sol-gel formaldehyde sensor

The selectivity of sol-gel sensor for the identification of formaldehyde in the system through the solution with similar structural formula and chemical properties. Both similar and different from formaldehyde, were studied within a closed system, namely methanol, acetaldehyde, benzaldehyde, toluene and acetone. From the result showed that the absorbance from the reaction between pararosaniline hydrochloride and formaldehyde provided the highest (Figure 7), indicating that this developed sol-gel sensor is most selective to formaldehyde.

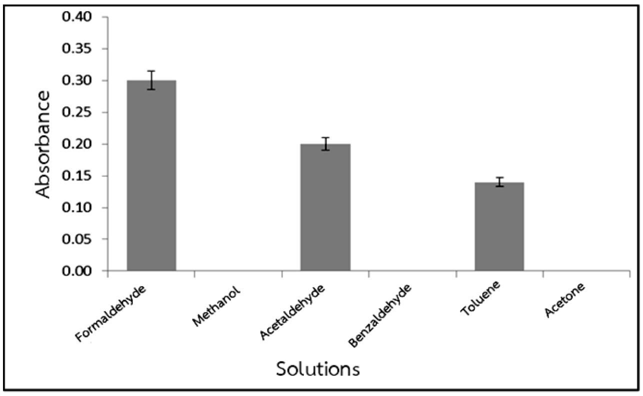

Figure 7 Sol-gel formaldehyde absorbance and other solutions.

Application of the sol-gel syringe chemical sensor for formaldehyde determination

In this research, we highlight the studied which have been successfully applied using mini-syringe sol-gel and sol-gel formaldehyde sensor detected formaldehyde of five shops in Yala Province, Thailand. Mini-syringe sol-gel formaldehyde sensor can be detected formaldehyde by observing the color change of sol-gel with the naked eye. Therefore, both of the mini-syringe sol-gel and sol-gel sensors were installed at the five shops namely A, B, C and D advertising shops and Furniture shop of E by installing formaldehyde sensor at 3 points inside of each shop. From the result found that the advertising shop A, C and D at point 1, which is the printer area, the formaldehyde concentrations of 6.60, 6.30 and 10.21 ppm, respectively. That means the amount detected exceeded the standards announced by the Ministry of Interior and the National Institute of Safety and Health in the United States. While the advertising shop B and furniture plywood shop E did not detected formaldehyde at all three points as shown in Figure 8.

Figure 8 The relationship between formaldehyde concentration and sample shops.
(Inset: The installing point of the mini-syringe formaldehyde sol-gel sensor inside the shop)

Discussion

The current work is mini-syringe sol-gel based colorimetric formaldehyde sensor with entrapped pararosaniline hydrochloride reagent to generate highly selective, portable and easily operated spectrometric procedure for the sensitive and selective of formaldehyde. The developed sensing device can be used as a qualitative with the naked eye as screening method for the real-time analysis of formaldehyde. Sol-gel formaldehyde colorimetric sensor offers substantial merits over the reported chromatography methods such as ignoring the pre-concentration process, eliminating the sophisticated procedures and excluding the harmful organic solvents. In addition, this work is distinguished by simplicity and laboratory availability. Therefore, mini-syringe sol-gel sensor using colorimetric spectroscopy as the transducer can be used as an alternative for the detection of formaldehyde in an indoor.

Conclusions and Suggestion

This research has shown that mini-syringe sol-gel sensors can be measured formaldehyde toxic gas. The developed formaldehyde sensor provided for a rapid screening test from the color changed with naked eye, highly selective, easy to use, inexpensive and portable. The sol-gel formaldehyde sensor was applied in the form of a mini-syringe to measure formaldehyde in air from the furniture and vinyl/poster shops in Yala Province, Thailand gave results that useful information in occupational health and safety. The research can also be further developed by composite nanomaterials with sol-gel sensors using electrochemical detection to increase analytical sensitivity, and so on, which has great potential for further commercial applications.

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