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doi:10.15199/48.2022.08.18

Agarose gel coated glass substrate for formaldehyde sensing application

***Abstract**. This paper demonstrated an intensity modulation detection system employing agarose gel coated on the glass substrate for formaldehyde sensing application. The objectives of this work is to develop a simple and low cost formaldehyde sensor using commercially available microscope glass substrate, coating material and data acquisition unit. The glass substrate was coated with agarose gel using low temperature synthesis method which has superiority in term of high porosity and capable to absorb molecule around it. The formaldehyde detection is based on the change in refractive index (RI) of the agarose gel as a coating material. The RI change of the coating materials will modulate the output light intensity when the concentration level of the formaldehyde varies. This is due to the intensity of the light weakening by absorption and scattering when light propagated through the sensing material. A significant response to formaldehyde concentrations was observed with the output voltage reduced linearly from 1.1V to 0.4V. The sensitivity and the linearity of the proposed sensor improve by a factor of 1.02 and 1.03 respectively as compared to uncoated glass substrate. Moreover, it performs better in term of stability, hysteresis and time response. The proposed formaldehyde sensor avoid utilization of costly optical sensor setup based on laser source which are not feasible for large scale production. Based on the experiment results, the proposed sensor has a good potential as a formaldehyde sensor which is essential for food, health and environmental sector.*

***Streszczenie.** W pracy przedstawiono system detekcji modulacji intensywności wykorzystujący żel agarozowy pokryty szklanym podłożem do zastosowania w czujnikach formaldehydu. Celem tej pracy jest opracowanie prostego i taniego czujnika formaldehydu przy użyciu dostępnego na rynku podłoża szklanego mikroskopu, materiału powłokowego i jednostki akwizycji danych. Podłoże szklane pokryto żelem agarozowym metodą syntezy niskotemperaturowej, która ma przewagę pod względem dużej porowatości i jest w stanie absorbować otaczające ją cząsteczki. Wykrywanie formaldehydu opiera się na zmianie współczynnika załamania światła (RI) żelu agarozowego jako materiału powlekającego. Zmiana RI materiałów powłokowych będzie modulować natężenie światła wyjściowego, gdy zmienia się poziom stężenia formaldehydu. Wynika to z intensywności osłabienia światła przez absorpcję i rozpraszanie podczas propagacji światła przez materiał czujnika. Zaobserwowano istotną odpowiedź na stężenia formaldehydu przy liniowo obniżonym napięciu wyjściowym z 1,1V do 0,4V. Czułość i liniowość proponowanego czujnika poprawiają się odpowiednio o współczynnik 1,02 i 1,03 w porównaniu z niepowlekanym podłożem szklanym. Ponadto działa lepiej pod względem stabilności, histerezy i odpowiedzi czasowej. Proponowany czujnik formaldehydu pozwala uniknąć kosztownych układów czujników optycznych opartych na źródle laserowym, które nie są możliwe do zastosowania w produkcji na dużą skalę. W oparciu o wyniki eksperymentu, proponowany czujnik ma dobry potencjał jako czujnik formaldehydu, który jest niezbędny w sektorze spożywczym, zdrowotnym i środowiskowym. (**Szklane podłoże pokryte żelem agarozowym do stosowania w czujnikach formaldehydu**)*

Keywords: formaldehyde, agarose gel, glass substrate
Słowa kluczowe: formaldehyd, żel agarozowy, podłoże szklane

Introduction

Formaldehyde is a colourless and strong-smelling gas which is mostly present in a water-based solution. Commonly, it is a natural chemical compound that usually used in food processing industry as a food preservative. However it could become a harmful element for human being if inappropriate amount used in foods processing [1]. It can be naturally found in foods for example fruits and veggies with a concentration of up to 300 to 400 mg/kg. There is no specific amount for daily consumption of formaldehyde for one person but World Health Organization (WHO) has estimated it to be between 1, 5 and 14 mg/d for average adult and according to European Food Safety Authority (EFSA) the formaldehyde consumption should not exceed 100 mg per day. The higher contains of this chemical compound can cause food poisonous and a lot of diseases such as damage to liver and kidney, leukaemia and cancer [1]. Furthermore, breathing problems, throat, nose, and eye irritation, coughing, nausea, sneezing, asthma, nasopharyngeal malignancy, nasal irritation, gastrointestinal tract, and esophagus and mouth inflammation are all possible adverse effects of excessive formaldehyde exposure [2, 3]. Because of its irritability, toxicity, and volatility, uncontrolled exposure to formaldehyde might be extremely detrimental to human health [4, 5]. As a result, research investigations for formaldehyde sensors with superior detection and monitoring capabilities are critical for human survival.

Optical sensing in liquid and gas has become an attractive approach for biochemical sensing, healthcare, food quality control, security, the environment, medication discovery, illness monitoring, biomolecular analysis, biomedical engineering, and environmental protection have all seen significant growth [6-8]. It has a number of benefits, including high precision, rapid reaction, lightweight, compact size, and immunity to electromagnetic interference. Due to the interaction between electromagnetic waves and biological/chemical molecules, the output signal of the sensors fluctuates [9, 10]. Utilization of glass substrate as a sensing platform exhibits several advantages such as a simple design, low-cost production and environmentally friendly device. The glass substrate is used to transmitted light with a certain incident angle and critical angle. As the light transmit through the sensing region of the glass substrate, transmission losses would happen. It experiences changes because behaviour of the light travel is defined by phase, intensity, frequency and polarisation [11]. As the glass substrates region exposed to the analyte, output voltage would change due to the interaction with the surround medium. The output voltage will affect the performance of the sensing device.

The sensing response of the glass substrate towards the surround medium is quite low due to small refractive index difference. In order to increase the sensing response, the glass substrate need to be coated with a higher refractive index such as agarose gel. Agarose gel is a hydrophilic material that exhibit high porosity which absorb molecule around it. The porosity decreases as the concentrations increase. It is based on swelling phenomenon which cause the refractive index of the gel change as the concentrations of the surrounding molecules change. A three-dimensional matrix made up of supercoiled agarose molecules collected into three-dimensional structures with channels and holes for biomolecules to flow through is known as a helical agarose gel. Because hydrogen bonds hold the 3-D structure together, it may be disrupted by reverting to a liquid state. Depending on the source, the melting temperature of an agarose gel differs from the gelling temperature. Agarose as the sensing element may absorb and expel moisture from or to the environment, changing its refractive index and capacity to adjust the intensity of light that passes through the proposed sensor. An agarose gel has a gelling temperature of 35–42 °C and a melting temperature of 85–95 °C [12].

Extensive studies on developing optical sensor based on various platforms such as aluminium foil [13], plastic optic fiber [14] , silica microfiber [15], PEN substrates [16], paper substrates [17], ITO/PET substrates [18] and polyimide substrates [19] have been demonstrated. However most of the platforms required complex design and high cost production. Furthermore, most recently optical detection devices utilize costly laser source, optical spectrum analyser and photodetector which are not feasible for large scale production [20]. A more practical sensor with low cost fabrication and manufacturing is required for mass production. This work reported the fabrication and integration of the formaldehyde liquid sensing device based on agarose gel coated glass substrate platform. This has been demonstrated for the first time to our knowledge. It comprises of simple and low cost light source and receiver to convert the output light that travel through the glass substrate to the voltage signal. As for output signal analysis, a conditioning amplifier and data acquisition unit based on arduino platform was used to avoid complicated and expensive equipment.

Sensing mechanism

The scattering coefficient of the coating material and the total fraction power carried in the sensing region have a significant impact on light travel through the glass substrate. The Lambert-Beer law, as given in equation (1), describes the light attenuation via the sensing medium [21]:

$$I = I_o e^{-\alpha L} \tag{1}$$

Where I is the intensity of light leaving the sensing region, I_o is the intensity of the light entering the sensing region, α is the scattering coefficient and L is the length of the sensing region. It also influenced by the absorbing material's bulk absorption coefficient, concentration, and effective fraction of total directed power [22]. When light propagates through the sensing material, as shown in Figure 1, the intensity of the light may be weakened by absorption and scattering. When light collides with an atom, scattering occurs. The atom is stimulated to a higher energy level, then returns to its previous level, emitting a photon with the same frequency as the one it received. When there are refractive index mismatches at borders, this happens. Extinction is the cumulative impact of these phenomena.

When the refractive index of the surround analyte increases, optical transmittance (T) decreases. This causes more light loss, which enhances the sensitivity to formaldehyde levels [23]. During exposure to varying concentrations of formaldehyde, the output intensity surrounding the detecting area varies [24]. Under varying concentration levels, the intensity of light throughout the absorbing medium changes. It corresponded to analyte concentration variations in the detecting area [21]. Equation (2) may be used to calculate optical transmittance (T) at the sensor's output [23]:

$$T = \frac{I}{I_o} = e^{-\alpha L} \tag{2}$$

Furthermore, variation of analyte concentrations would affect the sensing material due to the change in electrical conductivity that altered the complex refractive index which change the optical properties of the sensitive elements [25]. The output voltage decrease when the concentration level of the formaldehyde increase. It is due to the scattering effect of the coating layer when exposed to the different formaldehyde concentration level. Other factors are the variation of the surrounding refractive index and change in electrical conductivity due to adsorption process when analyte applied to the coating layer [26]. Different concentration level of formaldehyde will vary the output voltage produces. Higher formaldehyde concentration level will lower the output voltage due to light absorption around the sensing region. When formaldehyde concentrations increase, refractive index of the agarose coating layer become larger as compared to the glass substrates. This lead to greater light leakage when concentrations increase. Therefore, less light reach to receiver when concentrations increase. This decrease the output voltage from the proposed structure. The sensing mechanism is illustrated in Figure 1.

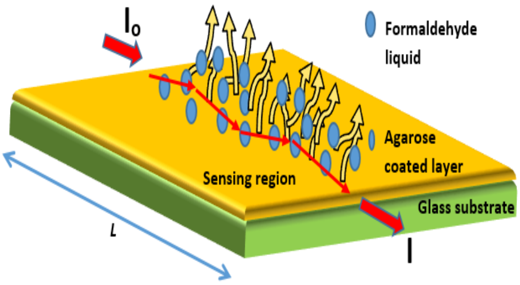


Fig.1. Sensing mechanism of the proposed sensor

Experiment details

Initially, the glass substrates need to be coated with agarose gel. The agarose gel was prepared by dissolving the agarose gel (Sigma Aldrich) into water. Then the mixture was heated to 50° Celsius. There are two types of method in preparation of agarose gel which is using the microwave and hot plate. The 0.5% weight content agarose gel has higher sensitivity than the 1% and 1.5% weight content agarose gel due to the effect of pore size study. The microwave agarose gel is normally weighted out in 1.5g agarose and combined with 100ml distilled water. The solution is then wrapped in microwaved for 1 minute and 20 seconds on high power. For the hot plate method, the agarose powder was dissolved in water in 0.5% weight quantities and being heated to 50˚C [12]. Subsequently, small aliquot of the mixture was deposited on the glass substrate and was left in normal room temperature for 24 hours. The agarose gel coated glass substrate is shown in Figure 2.

By referring to Figure 3, the experimental setup of the proposed sensor comprised of several components such as light source, receiver, conditioning amplifier, data acquisition system (DAQ) and personal computer (PC). For the light source, a green LED with central wavelength of 517.5 nm and bandwidth of 5nm was employed. Green LED was chosen for the light source based on previous study conducted by [14]. It also has advantages in term of optical and electrical properties for sensing applications [27].The position of the light source was place as close as possible to the edge of the glass substrate at an incident angle around 50° which result in refraction angle of the light inside the glass to be around 30°. Based on Snell's Law, this cause the light propagation experience total internal reflection which reducing the intensity losses. Therefore, the intensity losses occurred due to the scattering from the coating material [20]. As for receiver, photodetector is employed to convert the light signal to voltage signal. It is then connected to the conditioning amplifier to increase the output voltage from the receiver. A simple amplifier circuit comprise of LTI884 op-amp chip, 7.8 MOhm resistor and 10 pF capacitor is used to amplify the output voltage that is suitable for data acquisition unit [28]. A simple and low cost DAQ unit which is Arduino microcontroller is used in the proposed sensor. Subsequently, the DAQ is connected to the PC to perform control program and display the output signal for data analysis. The experiment was conducted in room temperature to emulate the real environment scenario. It was also performed inside control chamber to avoid the influence of other surround element. The experiment was repeated for formaldehyde concentration level from 0% to 100% in which the results were investigated with refer to 0% (pure water) [29]. The behaviour of the output voltage was then analysed according to the performance sensing parameters such as sensitivity, linearity, repeatability, hysteresis, stability and response time.

Fig.2. Agarose gel coated glass substrate

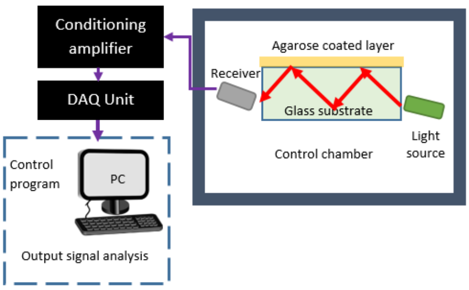
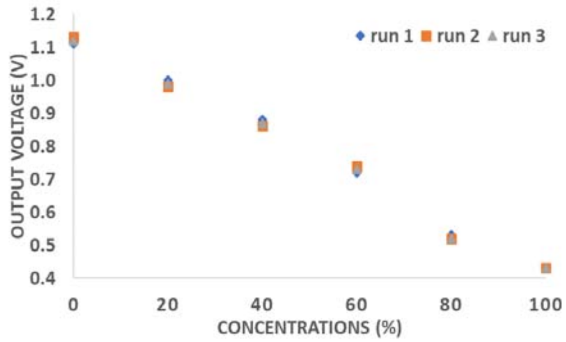


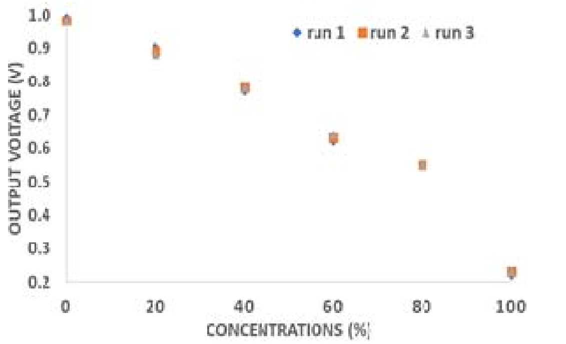
Fig.3. Experimental setup of the proposed sensor

Result and discussion

The repeatability of the output voltage for the uncoated glass substrate when exposed to difference formaldehyde concentrations level is depicted in Figure 4 (a) while Figure 4(b) shows the repeatability of the output voltage of the agarose gel coated glass substrate. The repeatability test reveals acceptable reproducibility results for both samples. While Figure 5 shows the hysteresis graph of both samples. It was conducted by recording the output voltage during forward and reverse measurement. Based on Figure 5 (a), the uncoated glass substrate produces quite large difference output voltage which is around 0.12V at 80% concentration level. The agarose coated glass substrate produces better hysteresis trend with smaller output voltage difference as shown in Figure 5 (b). This is owing to a minor variation in the sensor response due to a discrepancy in the rates of adsorption and desorption of analyte molecules on the coating layer [30]. In order to verify the sensor's functionality, stability test was performed by logging the output data for 10 minutes (600 seconds) in every second for five formaldehyde concentrations level. Figure 6 (a) shows the stability data for the uncoated glass substrate. The graph shows a lot of distortion as compared to the agarose coated glass in Figure 6 (b) which shows the proposed sensor has better stability.

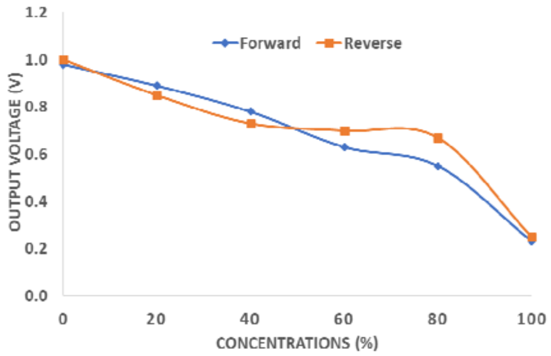


(a)

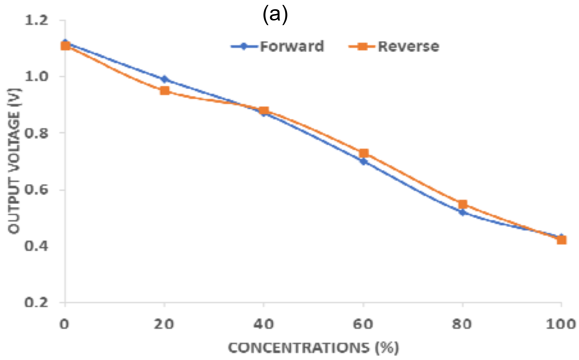


(b)

Fig.4 Repeatability graph for; a) Uncoated glass and b) Agarose coated
Glass



(a)



(b)

Fig.5. Hysteresis graph for; a) Uncoated glass and b) Agarose gel coated glass

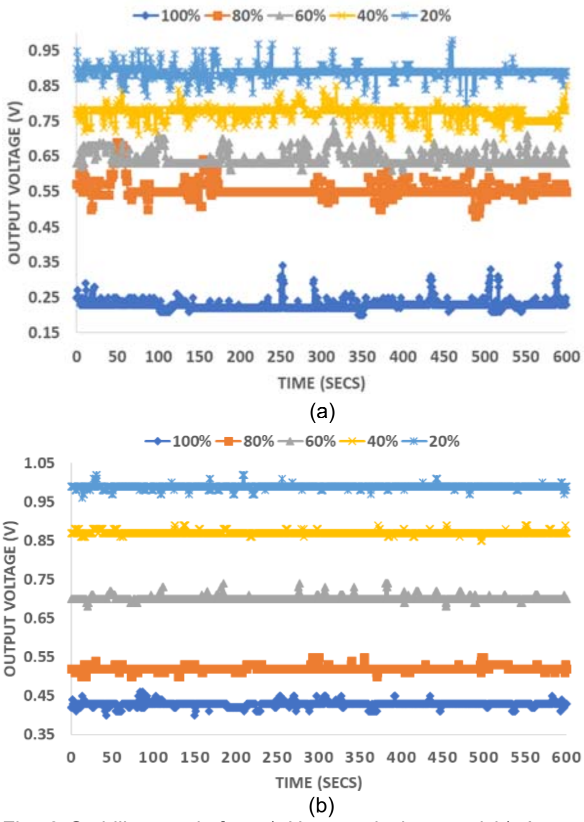


Fig. 6 Stability graph for; a) Uncoated glass and b) Agarose gel coated glass

Subsequently, the response time and the recovery time of the proposed sensor was investigated. It was conducted by applying the sensing element to the samples from minimum concentration value to maximum concentration value for response time and vice versa process for recovery time. Response time and recovery time of both samples are shown in Figure 7 (a) and Figure 7 (b) respectively. The uncoated glass takes around 0.5 seconds for both response time and recovery time which is slower as compared to the glass substrate coated with agarose gel with 0.3 seconds. This observation proved that a glass substrate coated with agarose gel responds and recovers faster than the uncoated glass substrate. Eventually the sensing response of the proposed sensor ranging from 0% to 100% concentrations level was investigated. Light propagation through the glass substrate attenuated due to both forward and backward scattering with the presence of formaldehyde analyte on the glass. Forward scattering dominates when the analyte concentrations increase which lead to the reduction of output voltage. The sensitivity of the uncoated glass is 0.007 V/% with linearity of 97.21 % while the glass substrate coated with agarose gel has the higher sensitivity of 0.0072 V/% with linearity of 99.66% as shown in Figure 8. This is due to the agarose gel coated glass substrate has higher refractive index of 1.3746 as compared to the uncoated glass substrate which cause a lossy waveguide which lead to the decrement of the output voltage [26]. The summary of the sensing performances is shown in Table 1.

Table 1. Summary Of The Sensing Performances

Parameters	Uncoated glass	Agarose coated glass
Sensitivity (V/%)	0.007	0.0072
Linearity (%)	97.21	99.66
Time response (Secs)	0.5	0.3

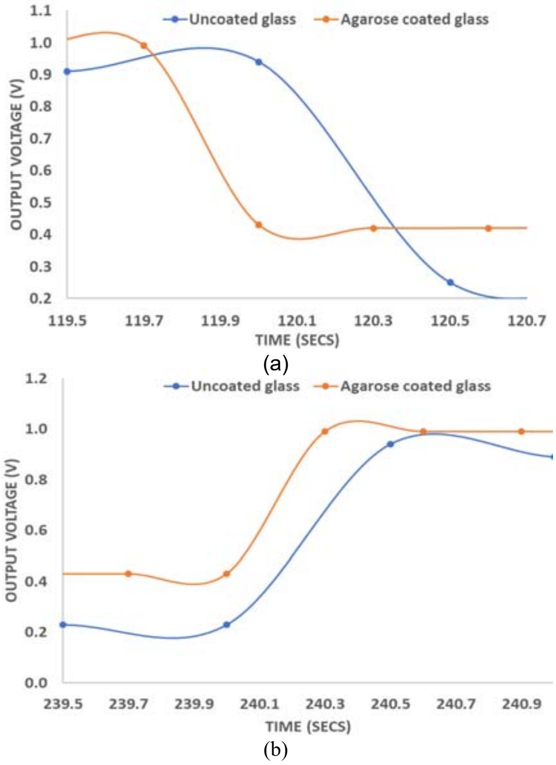


Fig. 7 Time response for both samples; a) Overall, b) Response time and c) Recovery time

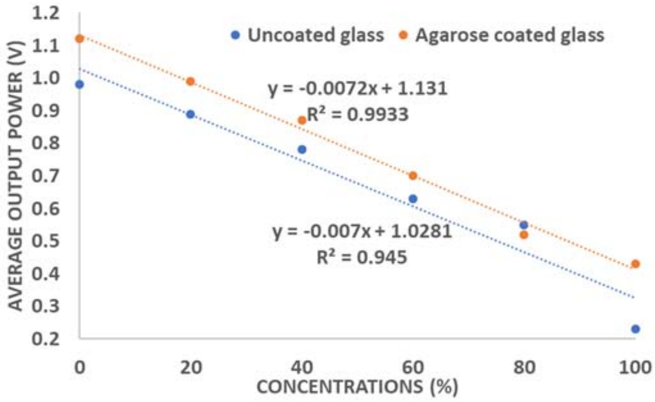


Fig.8 Trendline graph for both samples.

Conclusion

A formaldehyde sensor has been successfully demonstrated by fabricating low-cost and simple detection device. The proposed approach has advantages of evading the used of expensive laser source based equipment which is less practical for large scale production. The utilization of components which are widely available in the market make the proposed sensor a viable formaldehyde sensor. Overall the proposed sensor has shown better sensing performances results in term of sensitivity, linearity, response time, repeatability, stability and hysteresis as compared to its counterpart. This is due to the used of agarose gel coated material which enhanced the light interaction towards the variation of refractive index when the formaldehyde concentrations level increase. In future, numerous coating material could be applied as a sensitive material to investigate the sensing performance towards formaldehyde concentrations level

Acknowledgements
The authors would like to thank Universiti Teknikal Malaysia Melaka for their financial support.

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