

Optimization of light source wavelength for ammonia detection in water

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ABSTRACT

Optimization of light source wavelength for ammonia detection in surface water is presented in this work. For the ammonia detection, the surface water sample is mixed with sodium chloride and nessler reagent, whereas the sensor head consists of unclad plastic optical fiber. The unclad region has a length of 1 cm and the cladding is removed by immersing it in acetone solution. Experimental results indicate that the output light intensity of the sensor has linear relationship with the ammonia concentration. At the wavelength of 510 nm, the output light increases linearly as the ammonia concentration varies from 0.07 mg/L to 8.97 mg/L. At the same wavelength, the proposed sensor achieves the sensitivity of $0.0139 \text{ (mg/L)}^{-1}$, accuracy of 99.59% and resolution of $0.72 \text{ }\mu\text{g/L}$. The analysis of light source wavelength reveals that a wavelength range from 450 nm to 580 nm produces the optimized performances. Within this wavelength range, the proposed sensor achieves sensitivity of higher than 0.01 (mg/L)^{-1} , accuracy of higher than 99% and resolution of less than $1 \text{ }\mu\text{g/L}$.

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1. INTRODUCTION

Ammonia has been widely used in various industrial processes, agricultural activity, and a variety of biological systems [1]. Ammonia molecules are a nutrient required for life, but excess ammonia will cause toxic effect on the health of plants, animals as well as human beings. Consuming water with presence of ammonia will corrode the lining of the mouth, esophagus, and stomach. Furthermore, excess ammonia level may affect aquatic life. Previous studies have found that lethal ammonia concentration for a variety of fish species ranges from 0.2 mg/L to 2.0 mg/L [2]. However, the ammonia tolerance varies between fish species and physiological status. To avoid the environmental risk, the Food and Agriculture Organization (FAO) has advised that the ammonia levels in water should be lower than 1 mg/L. Thus, it is essential to control the concentration of ammonia in water to prevent environmental pollution.

Recently, a variety of sensing techniques for measuring ammonia concentration in water have been suggested, such as electro-chemical method [3], metal oxide semiconductor detectors [4], a ratiometric fluorescence sensor [5], and fiber optic based sensors [2], [6]–[11]. The electrochemical method has high

selectivity and low detection limit. However, this method lacks in term of the lifetime, miniaturization, and stability of the reference electrodes [5]. On the other hand, metal oxide semiconductor detector has low detection limit with fast response time, but has low sensitivity and lack in selectivity [1]. Ratiometric fluorescence technique meanwhile has great advantages regarding high sensitivity and selectivity with low detection limit [5]. Nonetheless, this method has complex acquisition and data manipulation due to the use of fluorescence ratio [12]. Fiber optic based sensor, on the other hand, is widely known for its immunity to electromagnetic interference, small in size, and ruggedness. Furthermore, fiber optic sensor has low loss, low dispersion, ultrawide bandwidth, high dynamic range, durability, upgradability, and low cost [9].

The basic operating principle of the fiber optic sensor is that the light signal travelling through an optical fiber changes when it is subjected to chemical or physical stimulus [13]. Such mechanism allows the fiber optic sensor to find applications in detecting properties such as temperature [14], [15], liquid level [16], [17], refractive index [18]–[20], uric acid concentration [21], [22] and ammonia concentration [2], [6]–[11]. For ammonia detection in water, many methods have engineered the fiber such that a part of the cladding of the fiber sensor is removed through chemical etching, tapering or a combination of etching and tapering. In order to enhance the sensor sensitivity and selectivity in detecting ammonia concentration, additional coating material such as tin dioxide [2], oxazine 170 perchlorate [6]–[8], sol-gel silica [9], [10] and zinc oxide [11] were deposited on the modified cladding. However, such developments [2], [6]–[11] have limitations in that they lack analysis of the light wavelength for the optimized performances for ammonia detection in water. In this work, the light source wavelength is analysed experimentally for the optimized performances with respect to the sensitivity, accuracy and resolution.

2. EXPERIMENT

2.1. Experimental setup

The ammonia concentration in surface water samples is detected using clad modified fiber optic sensor where the cladding of the optical fiber is removed using etching method. The optical fiber used is a 15 cm long plastic optical fiber (POF) with polymethyl methacrylate (PMMA) core. The POF has a diameter core of 980 μm and diameter cladding of 20 μm thick. The refractive index of the core and cladding are 1.492 and 1.417, respectively. The sensing region of the fiber is prepared based on chemical etching technique using acetone, de-ionized water, and sandpaper. No additional coating is deposited on the modified cladding surface. The sensor head of the POF is prepared by removing 1 cm of the middle fiber jacket using a fiber stripper. The cladding of the sensor head is then removed by immersing the optical fiber sensing region in acetone solution for 10 seconds. Reaction between acetone and the cladding forms a milky white foam on the outer fiber surface. To neutralize this reaction towards acetone, the fiber is then dipped in de-ionized water. The milky white foam on the outer fiber surface is then removed using 2000 grit sandpaper and cleaned again using de-ionized water. The unclad part of the fiber (sensing region) is then tested using FIS fiber optic continuity tester as shown in Figure 1(a). The test is conducted to ensure perfection on the sensor head. The POF is then connected to ocean optics deuterium halogen (DH)-mini light source, and ocean optics HR4000CGUV-VIS spectrometer for measurement as shown in Figure 1(b).

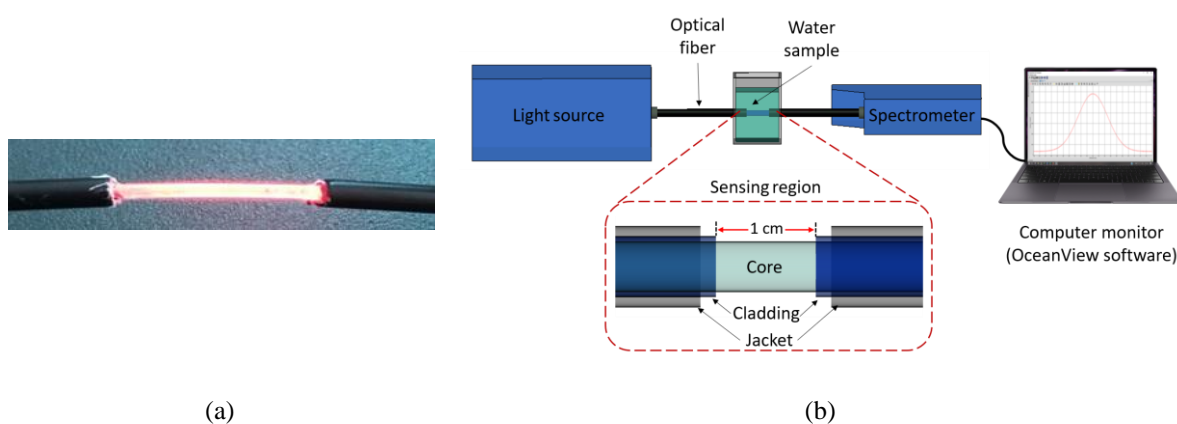


Figure 1. Experimental setup of fiber optic sensor that consists of (a) unclad POF as sensor head and (b) light source and spectrometer

2.2. Sample preparation

The detection of ammonia in this work is based on the reaction of surface water sample with two reagents: sodium chloride (NaCl) and nessler (K_2HgI_4). The surface water samples are collected in the area of Batu Pahat, Johor, Malaysia. The sample of the surface water collected is shown in Figure 2(a). The water sample used for measurement is prepared by mixing 1 mL of surface water sample with 9 mL of sodium chloride reagent as shown in Figure 2(b). Then, 4 drops of nessler reagent are added into the mixture. The addition of nessler reagent causes a brown precipitate in the sample as shown in Figure 2(c). The refractive index and concentration of each sample is measured using Atago refractometer and Hanna instrument ammonia high range portable photometer (HI96733), respectively. The concentrations obtained are 0.07 mg/L, 2.63 mg/L, 6.93 mg/L, and 8.97 mg/L. Such concentrations act as the reference for the result analysis. For the measurement, 1.5 mL of the sample is added into a cuvette as shown in Figure 1(b). The light intensity of each sample is observed and recorded using OceanView software installed in a computer. Each water sample is tested 3 times and the average value of each concentration is obtained subsequently.

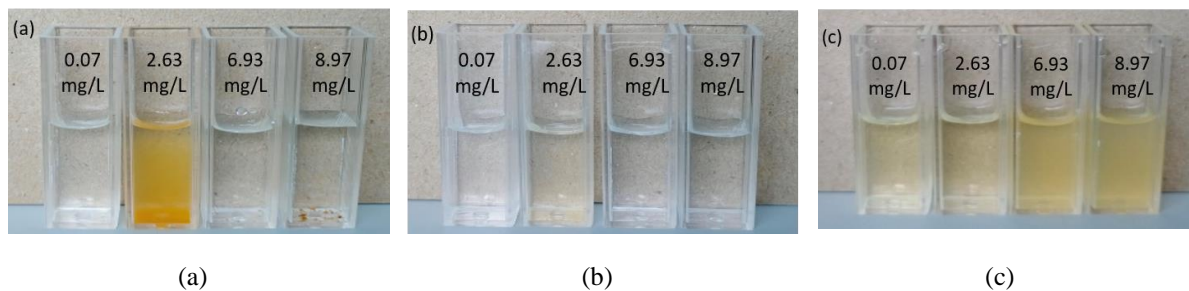


Figure 2. Preparation of water sample for testing: (a) water sample only, (b) water sample with sodium chloride reagent, and (c) water sample with sodium chloride and nessler reagent

3. RESULTS AND DISCUSSION

Table 1 shows the refractive index of the sample against the ammonia concentration. Based on Table 1, it is evident that the refractive index of the sample increases when the ammonia concentration in the sample increases. Meanwhile, Figure 3 illustrates the spectrum of light intensity against wavelength with different concentrations of ammonia. Based on Figure 3, it is found that the light intensity increases as the ammonia concentration in the sample increases. This condition is related to the increase of refractive index with the ammonia concentration shown in Table 1. As the refractive index of the sample increases, the refractive index difference between the sample and fiber core reduces. Consequently, the light loss is reduced, causing the output light intensity of the sensor becomes higher [23], [24].

Table 1. Refractive index of water sample

Concentration (mg/L)	Refractive index
0.07	1.3322
2.63	1.3330
6.93	1.3340
8.97	1.3348

For the case of study, the change of light intensity towards different ammonia concentrations at the wavelength of 510 nm is presented in Figure 4. The graph is obtained based on the data obtained from Figure 3. Based on the data shown in Figure 4, it is found that the output light intensity has linear relationship with the ammonia concentration. The sensitivity of the sensor is $0.0139 \text{ (mg/L)}^{-1}$, which is obtained based on the slope of the linear graph [23]. On the other hand, the sensor shows accuracy of 99.59%. The accuracy of the sensor is calculated using the (1) [21].

$$\text{Accuracy} = 1 - |(c_{real} - c_{measured})/c_{real}| \quad (1)$$

Where c_{real} is the real ammonia concentration measured by a commercial hanna instrument ammonia high range portable photometer (HI96733) and $c_{measured}$ is the measured concentration obtained from the linear fit equation in Figure 4. Meanwhile, the sensor has resolution of $0.72 \mu\text{g/L}$, which is calculated using the following the (2) [25].

$$Resolution = \frac{n}{S} \quad (2)$$

Where n is the smallest scale that can be measured by detector and S is the sensitivity of the sensor. In order to determine the best wavelength range for ammonia detection, the sensitivity, accuracy and resolution of the fiber optic sensor is calculated for every 10 nm wavelength from 400 nm to 900 nm. The data obtained is presented in Figure 5.

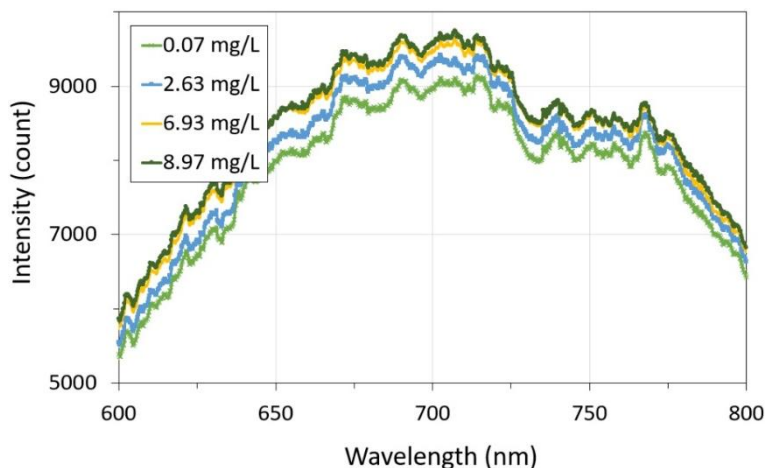


Figure 3. Light intensity as function of wavelength for different ammonia concentration

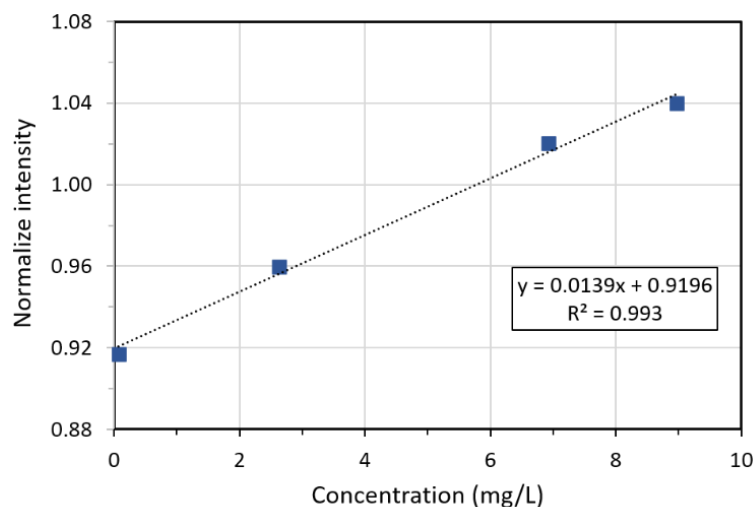


Figure 4. Light intensity as function of ammonia concentration at wavelength of 510 nm

Figure 5(a) plots the sensor sensitivity as a function of wavelength. It can be seen that the sensitivity is higher than 0.01 (mg/L)^{-1} at a wavelength range from 440 nm to 580 nm. At wavelength ranges from 400 nm to 430 nm and 590 nm to 900 nm, the sensor has sensitivity of less than 0.01 (mg/L)^{-1} . As for the accuracy as illustrated in Figure 5(b), the accuracy of the system is higher than 99% when the wavelength is between 450 nm and 900 nm. Whereas the sensor possesses less than 99% accuracy as the wavelength spans from 400 nm to 440 nm. On a different note, the sensor has resolution of less than $2 \text{ } \mu\text{g/L}$ at a wavelength range from 430 nm to 900 nm as shown in Figure 5(c). Resolution of higher than $2 \text{ } \mu\text{g/L}$ is recorded when the wavelength spans from 400 nm to 420 nm. All in all, based on the findings in Figure 5, the best wavelength range for the optimized performances with respect to the sensitivity, accuracy and resolution is from 450 nm to 580 nm. Within this wavelength range, the sensor produces high sensitivity, accuracy and resolution.

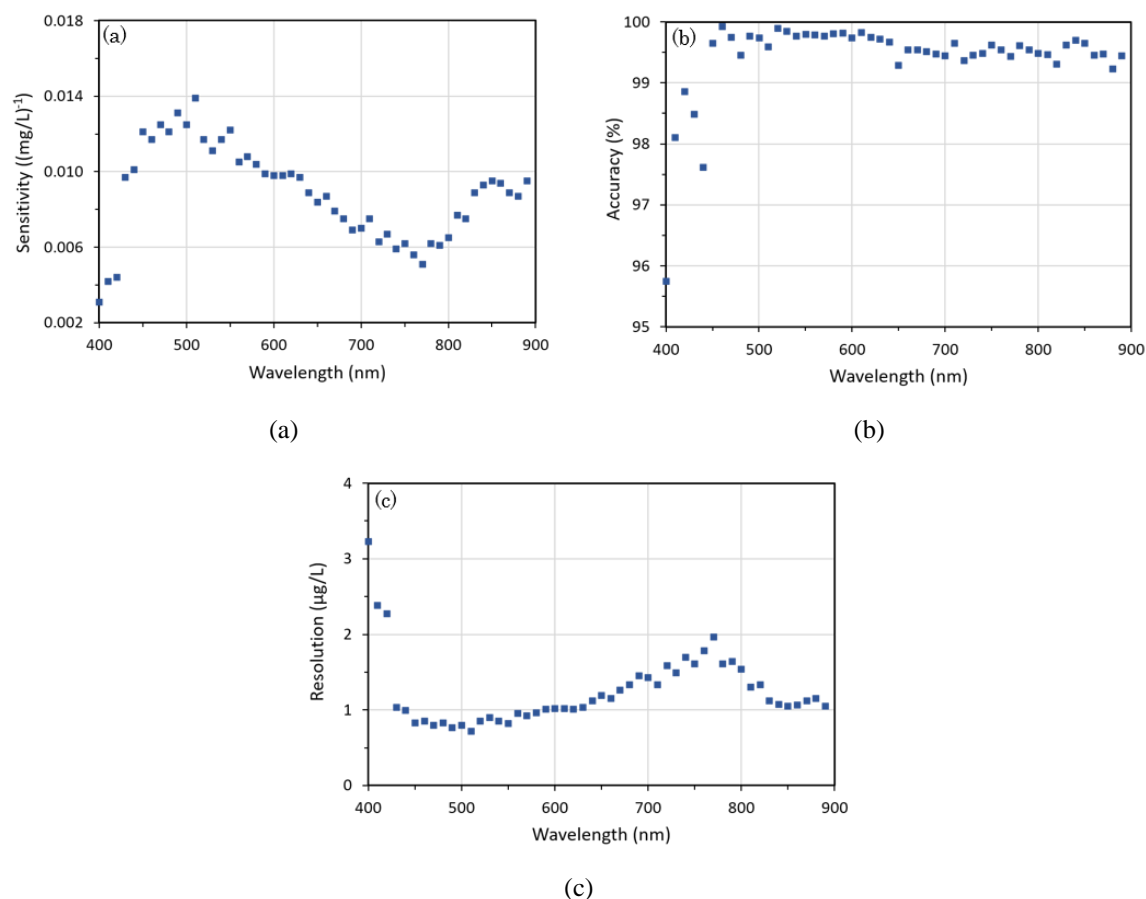


Figure 5. Performance analysis for light source wavelength range (400 nm – 900 nm) in terms of (a) sensitivity, (b) accuracy, and (c) resolution

4. CONCLUSION

In conclusion, optimization of light source wavelength for ammonia detection in the surface water is experimentally demonstrated in this work. The ammonia content in the water is detected by unclad plastic optical fiber with the presence of sodium chloride and nessler reagent. Based on experimental results, it is found that the output light intensity has linear relationship with the ammonia concentration. Experimental results suggest that the wavelength range that produces the optimized performances spans from 450 nm to 580 nm. Within this wavelength range, the proposed sensor achieves sensitivity of higher than 0.01 (mg/L)^{-1} , accuracy of higher than 99% and resolution of less than $1 \mu\text{g/L}$.

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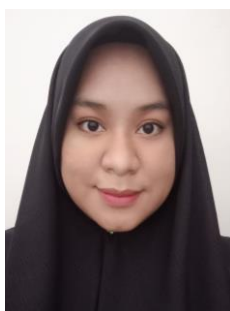
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Optimization of light source wavelength for ammonia detection in water (Nurfatihah Che Abd Rashid)






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