Evaluation of the tapered PMMA fiber sensor response due to the ionic interaction within electrolytic solutions

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Evaluation of the tapered PMMA fiber sensor response due to the ionic interaction within electrolytic solutions

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A tapered plastic multimode fiber (PMMA) optical sensor is proposed and demonstrated for continuous monitoring of solutions based on different concentration of sodium chloride and glucose in deionized water. The tapered PMMA fiber was fabricated using an etching method involving deionized water and acetone to achieve a waist diameter and length of 0.45 mm and 10 mm, respectively, and was used to investigate the effect of straight, U-shape, and knot shape against concentration for both sodium chloride and glucose. The results show that there is a strong dependence of the electrolytic and non-electrolytic nature of the chemical solutions on the sensor output. It is found that the sensitivity of the sodium chloride concentration sensor with the straight tapered fiber probe was 0.0023 mV/%, which was better than the other probe arrangements of U-shape and knot. Meanwhile, the glucose sensor performs with the highest sensitivity of 0.0026 mV/wt % with the knot-shaped tapered fiber probe. In addition, a tapered PMMA probe which was coated by silver film was fabricated and demonstrated using calcium hypochlorite (G70) solution. The working mechanism of such a device is based on the observed increment in the transmission of the sensor that is immersed in solutions of higher concentration. As the concentration varies from 0 ppm to 6 ppm, the output voltage of the sensor increases linearly from 3.61 mV to 4.28 mV with a sensitivity of 0.1154 mV/ppm and a linearity of more than 99.47%. The silver film coating increases the sensitivity of the proposed sensor due to the effective cladding refractive index, which increases with the coating and thus allows more light to be transmitted from the tapered fiber.

Keywords: polymethyl methacrylate (PMMA); tapered multimode fiber sensor; glucose; sodium chloride; calcium hypochlorite; electrolyte; non-electrolyte

1. Introduction

Optical fibers are currently available in abundance for various applications, including transmission media, which cover a wide spectral range, and sensors. Fiber sensor studies are focusing on many new areas, such as gas and vapor sensing, medical and chemical analysis, molecular biotechnology, marine and environmental analysis, industrial production monitoring, bioprocess control, salinity measurement and applications in the automotive industry [1–6]. Fiber sensors can be classified in terms of two main approaches: direct or indirect. Direct sensing is commonly used to measure intrinsic optical properties, e.g. refractive index, emission, or absorption [2,3]. Recently, tapered optical fibers have attracted much interest, especially for sensing applications [2]. This is due to the higher portion of evanescent field traveling inside the cladding in the tapered fiber and thus the traveling wave characteristics becoming more sensitive to the physical ambience of its surrounding. The interest in tapered multimode fibers has also increased since they are mechanically stronger and also easier to manufacture compared to single-mode fibers. Therefore, the tapered multimode fibre was demonstrated previously as a chemical sensor with a simple setup [4].

Most studies of optical-based chemical sensors have been based on the refractive index variation of the chemical under scrutiny [7,8]. However, the conductive nature of chemical solutions also plays a role in the output trend of the sensor [9]. Chemicals can be classified as electrolytes and non-electrolytes depending on the dissociation of their ions in solution [10]. An electrolyte is defined as a substance containing free ions that make the substance electrically conductive. A high proportion of solute dissociates to form free ions in strong electrolytes. Common types of electrolytes are group I and II salt solutions when in the form of ionic solution or molten. Electrolytes are important minerals in the human body which affect the amount of water in the body, blood acidify (pH), and muscle function [11]. Non-electrolytes are compounds composed of molecules that do not conduct
electricity when dissolved in molten or aqueous solutions. Non-electrolytes do not ionize in aqueous solution into positive and negative ions and hence fail to work as a conductor. They are normally covalent compounds and mainly organic in nature. Glucose, an example of a non-electrolyte, is important to all cells from bacteria to humans as a primary source of energy, in photosynthesis, and as fuel for cellular respiration [1,4].

In our earlier work, a simple intensity-modulated displacement sensor was proposed for sensing salinity based on different concentrations of sodium chloride in deionized water [2]. In this paper, a chemical concentration sensor is proposed and demonstrated using a tapered polymethyl methacrylate (PMMA) fiber. The main purpose of this study was to determine the effect of electrostatic charge on the fiber. Hence, sodium chloride and glucose were chosen in this experiment because of their solubility in deionized water and different degrees of ionic dissociation. The positively charged ions will be attracted to the negative surface of the tapered PMMA fiber via electrostatic interaction. This effect of the interaction on the sensor performance was studied by varying the concentration of sodium chloride and glucose solution, which is directly related to the number free ions in the solution.

The aims of this paper were to evaluate the performance comparison of the proposed sensor with various probe arrangements in sodium chloride and glucose solutions as examples of an electrolyte and a non-electrolyte, respectively, and also to study how a tapered fiber with silver coating can enhance the performance of the sensor. As the main component to free water from bacteria and contaminants is chlorine, here we are trying to construct a surface plasmon resonance (SPR) fiber sensor to measure the amount of chlorine concentration levels in drinking water and swimming pools. Accurate amounts of chlorine in water are essential to avoid exposure to high levels of chlorine that could cause respiratory system problems and/or irritation.

In order for a swimming pool to maintain its water disinfection levels, the chlorine concentration should be not more than 2 ppm [12].

The proposed sensor measures the output voltage of the detector that is influenced by the interaction of the evanescent wave produced in the tapered cladding and the solution which forms its surrounding. This technique offers simplicity, reliability, and continuous measurement capability [13]. The physical configuration of the optical fiber and surface charge interaction between fiber and solutions (sodium chloride and glucose) significantly affects the sensitivity of the fiber to an evanescent field [9]. Besides, a tapered fiber coated with a thin silver film, for application in measuring the concentration of calcium hypochlorite (G70), which is the chlorine added to a swimming pool, changes its optical properties in response to an external stimulus. This measurement is based on the intensity modulation technique, where the output voltage of the transmitted light is investigated for different chlorine concentrations.

2. Experiment
In the first part of the experiment, the plastic multimode fiber (PMMA) is tapered using a chemical etching technique. The PMMA used has an overall cladding diameter of 1 mm, a numerical aperture of 0.51, and an acceptance angle of 61°. The refractive index of the core and cladding are 1.492 and 1.402, respectively. Acetone, deionized water, and sand paper were used to taper the fiber. The acetone solution was applied to the PMMA fiber using a cotton bud. The PMMA fiber was then neutralized with the deionized water. The milky white surface around the outer cladding of the plastic fiber was removed using sand paper. This process was repeated until the tapered fiber had a stripped region length of 10 mm on average and waist diameter 0.45 mm. Finally, the tapered PMMA fiber was cleaned again using deionized water. Figures 1(a) and 1(b) show microscope

Figure 1. Microscopic image of the (a) un-tapered PMMA fiber (with diameter of 1 mm), (b) tapered PMMA fiber (with diameter of 0.45 mm), and (c) tapered PMMA fiber coated with silver film. (The colour version of this figure is included in the online version of the journal.)
images of the un-tapered and tapered PMMA fibers, which had a cladding diameter of 1 mm and 0.45 mm, respectively.

Figure 2 shows the experimental setup for sensing different chemical concentrations using the fabricated tapered PMMA fiber. The setup consists of a light source, an external mechanical chopper, a PMMA tapered fiber, a highly sensitive photo-detector, a lock-in amplifier, and a computer. The input and output ports of the tapered fiber are connected to the laser source and photo-detector, respectively. The light source used in this experiment is a He–Ne laser operated at a wavelength of 633 nm with an average output power of 5.5 mW. It was chopped at a frequency of 113 Hz by a mechanical chopper so as to avoid harmonics from the line frequency, which is about 50 to 60 Hz. The 113 Hz frequency was chosen as an odd number to prevent multiplication of 50 and 60 Hz; besides, it provides an acceptable value of output and stability. In addition, an increase to the value of chopper frequency causes the output voltage and stability to decrease. The He–Ne light source was launched into the tapered PMMA fiber placed in a Petri dish filled with the test solution. The output light was sent into the silicon photo-detector (818 SL, Newport) and the electrical signal was fed into the lock-in amplifier (SR-510, Stanford Research System) together with the reference signal of the mechanical chopper. The response time taken by the sensor to reach a stable response was 0.3 s, which corresponds to the post-processing time of the lock-in amplifier. The output result from the lock-in amplifier was connected to a computer through a RS232 port interface and the signals were processed using Delphi software. The reference signal from the chopper will match the input electrical signal from the photodiode. This allows a very sensitive detection system that will remove the noise generated by the laser source, photo-detector, and the electrical amplifier in the photo-detector.

In the experiment, the performance of the proposed sensor was investigated for two different chemicals: sodium chloride and glucose solutions as a representatives of an electrolyte and a non-electrolyte, respectively. The sodium chloride and glucose were dissolved in deionized water to prepare test solutions with various concentrations. Figure 3 shows the refractive index of the sodium chloride and glucose solutions (as measured by using a Mettler Toledo RE40D refractometer) against the concentration. As the concentration of sodium chloride increased from 0% to 12%, the refractive index of the solution also increased from 1.3325 to 1.3453, whereas the refractive index of glucose increased from 1.3325 to 1.3499.

At first, the PMMA tapered fiber in the Petri dish was straight aligned with respect to the detector to minimize bending losses that may occur in the tapered fiber. The tapered fiber, which acts as a sensor probe, was immersed in both solutions at various concentrations ranging from 0 wt % to 12 wt %. The experiment was then repeated for other cases where the straight tapered fiber is bent to form a U-shape and a knot shape for comparison purposes. During the experiment, the errors caused by temperature were taken to be negligible and the temperature was kept constant at 25°C.

In the second part of the experiment, an SPR fiber sensor was fabricated where a thin silver film was coated onto the tapered fiber by the electron-beam evaporation.
method using an Edward Auto 306 E-beam evaporator. The pressure deposition chamber was set to $2.1 \times 10^{-5}$ mbar, then filament voltage and current were set to 3.2 kV and 20 mA, respectively. To make sure that the fiber was coated evenly across its cylinder shape, both sides of the fiber were coated. This was done to make sure both sides of the sensing layer had a homogeneous metallic layer on them. After a constant deposition rate was established, the shutter was open for 20 s (around 20 nm), closed for a few minutes to allow cooling, and again opened for 20 s (around 20 nm). The two-step coating avoided any damage of the plastic fiber due to longer exposure. The measured thickness of the coated thin film was 40 nm, as measured using a Dektak surface profiler.

Figure 1(c) shows the tapered fiber coated with silver film. The image shows that there is a layer of shiny silver coated on the waist of the tapered fiber. Next, the experimental setup in Figure 2 was used to investigate the performance of the proposed sensor for various chlorine hypochlorite (G70) concentrations where the temperature was kept constant at 25°C. The experiment was carried out for both bare and coated tapered fibers.

Throughout the experiment, a fixed quantity of liquid solution was placed in the Petri dish and the corresponding output voltage was measured by a lock-in amplifier, which provides accurate measurements even though the signal is relatively very small compared to noise. Furthermore, a well-regulated power supply was used for the He–Ne laser and this minimized any fluctuation of source intensity.

3. Results and discussion

An experiment was carried out to study the variation of the transmitted power from the tapered PMMA fiber for different sodium chloride solutions, as presented in Figure 4. The figure shows that as the concentration of sodium chloride increases, the output voltage from the photo-detector also increases for straight, U-shaped and knot shape tapered fibers. It gives a linear increase in output voltage against concentration. The reduction of the fiber size increases the evanescent field penetration of guided modes [14]. Fibers made from PMMA will have a net negative charge at the surface of the fiber [9]. The fiber interacts with sodium chloride, which is a strong electrolyte, to attract the positive ions due to electrostatic charge and form a thin layer, which enlarges the pathway for the signal to pass through. The sensitivity of the fiber is influenced by the interaction length, bending effect, and electrostatic interaction between different concentrations of solution and the PMMA [9]. The highest sensitivity is obtained at 0.0022 mV/% and the slope gives a good linearity of more than 99% for a 0.96% limit of detection for the straight tapered fiber. The limit of detection was calculated by dividing the standard deviation by the sensitivity to show that the system is more efficient. It is noted that the straight, U-shape, and knot shape versions of PMMA tapered fiber sensor each experience an increase in voltage due the number of ions in the solution, which enables it to be more conductive [2,4,10]. The bending effect of fiber induces loss in both the U-shaped and knot-shaped tapered fiber. The highest loss is obtainable with the knot-shaped tapered fiber as more bending loss occurs for this type of configuration [6–8].

The same setup as in Figure 2 was used to repeat the experiment on the variation of the transmitted power from the tapered PMMA fiber for different glucose solutions, as presented in Figure 5. The figure shows that as the concentration of glucose increases, the output voltage from the photo-detector decreases for all tapered fiber configurations. It gives an inversely proportional relationship of output voltage against concentration.

Figure 4. Output voltage versus sodium chloride concentration. (The colour version of this figure is included in the online version of the journal.)
This is because glucose is a non-electrolyte where the conductivity of the solution decreases as the glucose concentration in the solution increases [10]. Glucose does not change into ions even though it dissolves in water and therefore the output is directly influenced by refractive index [4]. The sensitivity is obtained at 0.0026 mV/% and the slope gives a good linearity of more than 93% for a 0.56% limit of detection for the knot-shaped tapered fiber. The results show that the best performance is obtainable with the knot-shaped tapered fiber, with a sensitivity of 0.0026 mV/%, followed by the U-shaped tapered fiber, and, lastly, the straight tapered fiber. In this case, the bending effect boosts the sensitivity of the sensor as additional loss introduces further drop in the sensor output [6–8]. These results show that the proposed sensor is applicable due to the ability of the sensor to detect various amounts of ions continuously and the sensitivity is dependent on electrostatics interaction between solutions (sodium chloride and glucose) and the PMMA.

Table 1. The performance of the sodium chloride concentration sensor.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Straight</th>
<th>Bend</th>
<th>Knot</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sensitivity (mV/%)</td>
<td>0.0023</td>
<td>0.0017</td>
<td>0.001</td>
</tr>
<tr>
<td>Linear range (%)</td>
<td>0–12</td>
<td>0–12</td>
<td>0–12</td>
</tr>
<tr>
<td>Linearity (%)</td>
<td>&gt;99</td>
<td>&gt;98</td>
<td>&gt;99</td>
</tr>
<tr>
<td>Standard deviation (mV)</td>
<td>0.0022 (1.86%)</td>
<td>0.0017 (1.42%)</td>
<td>0.0013 (1.05%)</td>
</tr>
<tr>
<td>Limit of detection (%)</td>
<td>0.96</td>
<td>1.00</td>
<td>1.29</td>
</tr>
</tbody>
</table>

The performance of the sodium chloride concentration sensor is summarized in Table 1 for straight, U-shaped, and knot-shaped tapered fibers. The highest sensitivity was obtained at 0.0023 mV/% and the slope gives a good linearity of more than 99% for a 0.96% limit of detection for the straight tapered fiber. For the U-shaped tapered fiber, the sensitivity was obtained at 0.0017 mV/% and the slope gives a linearity of more than 98% for a 1.00% limit of detection, while for the knot-shaped tapered fiber, the sensitivity was obtained at 0.001 mV/% with a linearity of more than 99% for a 1.29% limit of detection. Overall, the sensor is observed to be sufficiently stable with a standard deviation of 1.86% for straight tapered fiber, 1.42% for U-shaped tapered fiber, and 1.05% for knot-shaped tapered fiber.

The performance of glucose concentration sensor is summarized in Table 2 for straight, U-shaped, and knot-shaped tapered fibers. The sensitivity was obtained at 0.0013 mV/% and the slope gives a good linearity of more than 92% for a 0.94% limit of detection for the straight tapered fiber. For the U-shaped tapered fiber, the sensitivity was obtained at 0.0015 mV/% and the slope gives a linearity of more than 97% for a 0.84% limit of detection, while for the knot-shaped tapered fiber, the sensitivity was obtained at 0.0026 mV/% with a linearity of more than 93% for a 0.56% limit of detection. Overall, the sensor is observed to be sufficiently stable with a standard deviation of 2.75% for straight tapered fiber, 1.5% for U-shaped tapered fiber, and 0.095% for knot-shaped tapered fiber. These values were recorded for 100 s.

Figure 6 shows the variation of the transmitted light from the tapered fiber against the concentration of chlorine solution with and without silver film deposited onto the tapered region. The transmitted light intensity is...
observed to be higher with silver film coating since the silver has a much lower refractive index, which is about 0.134 at a wavelength of 633 nm [15], compared to water and this is probably cause by the reflection of silver. With the silver, the effective cladding refractive index decreases and thus more light is allowed to be transmitted in the tapered fiber. As shown in Figure 6, the output voltage from the photo-detector, which corresponds to the transmitted light intensity, increases linearly as the concentration of the G70 solution increases. Without the coating, the sensitivity of the sensor is obtained at 0.0029 mV/ppm with a slope linearity of more than 94.67% and limit of detection of 2.28 ppm. Meanwhile, the sensor with silver film coating produces a better sensitivity of 0.1154 mV/ppm with a better slope linearity of more than 99.47% and a limit of detection of 0.029 ppm.

Since the diameter of the cladding of the tapered fiber has been reduced, the refractive index of the surrounding medium works as passive cladding and can influence the amount of power loss as the signal propagates through the tapered region. When immersing the tapered fiber into the chlorine solutions with various concentrations ranging from 0 ppm to 6 ppm, the refractive index of the surrounding medium increases since the refractive index of chlorine is larger than water (1.333). Since the refractive index of the composite increases as the concentration increases, the core and cladding index difference for the proposed sensor reduces with the increment of concentration. Therefore, less light than that propagating inside the tapered region leaks to the surroundings, which results in the output voltage increment. That is why when concentration of chlorine solution increases, output voltage increases for both fiber with and without silver coating.

From Figure 6, it can be seen that the sensitivity of the tapered fiber was enhanced when coated with silver thin film compared to the bare fiber. This is due to the surface plasmons wave excitation that happens at the metal–dielectric surface. When monochromatic light is incident into the fiber, input rays undergo reflections at the boundary of metal and dielectric with an SPR coupling angle. At this angle, optical power, which is the intensity of light, is attenuated as the energy of incident light power now being transferred into an SPR charge density wave. So, the refractive index at the sensing region will change with concentration of the chlorine sample, resulting in a change of optical power measurement, which is the output voltage measured in the experiment [16].

The performance characteristics of the proposed sensor are summarized in Table 3. Overall, the sensor is observed to be sufficiently stable with standard deviations of 0.0066 mV (0.37%) and 0.0033 mV (0.084%) for PMMA probe without and with silver thin film. as recorded for the time duration of 100 s. These results show that the proposed sensor is applicable and useful for the detection of calcium hypochlorite (G70), a

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Straight</th>
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<th>Knot</th>
</tr>
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<tbody>
<tr>
<td>Sensitivity (mV/%)</td>
<td>0.0013</td>
<td>0.0015</td>
<td>0.0026</td>
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<td>Linear range (%)</td>
<td>0–12</td>
<td>0–12</td>
<td>0–12</td>
</tr>
<tr>
<td>Linearity (%)</td>
<td>&gt;92</td>
<td>&gt;97</td>
<td>&gt;93</td>
</tr>
<tr>
<td>Standard deviation (mV)</td>
<td>0.0012 (2.75%)</td>
<td>0.0012 (1.5%)</td>
<td>0.0015 (0.95%)</td>
</tr>
<tr>
<td>Limit of detection (%)</td>
<td>0.94</td>
<td>0.84</td>
<td>0.56</td>
</tr>
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</table>

Figure 6. Output voltage versus calcium hypochlorite (G70) concentration for the proposed tapered PMMA based sensor with and without silver thin film. (The colour version of this figure is included in the online version of the journal.)
common agent for sanitizing drinking water and swimming pools due to its ability to form hypochlorous acid to kill the bacteria, viruses, and contaminants in the water [17]. The 1–6 ppm sample range was chosen as a means of covering the amount of chlorine commonly found in swimming pools, which is around 0.5 ppm to 1.5 ppm. The high sensitivity demonstrated by this sensor enables the detection of any lower chlorine concentration. In addition, the lower limit of detection of coated silver compared to bare fiber (without silver) shows that the system is more efficient. The limit of detection is calculated by dividing the standard deviation by the sensitivity, which indicates that the system is more efficient when the value is lower.

The thickness of the silver coating should be optimum for SPR excitation. If the coating is too thick, signal loss will occur as the evanescent wave decays before reaching the analyte. However, if the coating is too thin the coupling efficiency is not optimum for SPR excitation [18].

### 4. Conclusions

In this work, we have successfully evaluated the performance of different configurations of tapered plastic fibers for different concentrations of sodium chloride and glucose solutions. These test solution have been chosen as representatives of an electrolyte and a non-electrolyte, respectively. Based on the results, the dissociation ability of the ions within the test solution plays an important role on the output performance. The evanescent field absorption in surrounding solutions of a PMMA tapered fiber is not only governed by the absorption coefficient and refractive index of the solution. The sensitivity of the fiber to evanescent field absorption is also significantly affected by the physical configuration of the optical fiber itself, as well as surface charge interactions between the fiber and the molecules/ions in the solution. It is believed that this information will be valuable for researchers in tailoring the best tapered fiber configuration with their test solution.

On the other hand, a simple chlorine sensor is proposed and demonstrated using a tapered PMMA coated with silver thin film for measurement of different concentrations of calcium hypochlorite (G70) in deionized water. As the concentration of chlorine increases, the index difference between core and cladding of the tapered fiber reduces and thus increases the transmissivity of the tapered PMMA. The silver thin film coating increases the sensitivity due to SPR nature and allows more light to be transmitted from the tapered fiber. Future works will confirm the SPR effect of silver coated on bare fiber. The proposed sensor provides numerous advantages, such as simplicity of design, low cost of production, higher mechanical strength, and ease of handling compared to silica fiber optics. Results show that the silver-coated fiber exhibits an increase in conductance of the fiber, and is thus applicable as an electrical chemical sensor.

### Funding

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