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A simple method for detection of low concentrations of fluoride in drinking water

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ABSTRACT

Naturally occurring elevated levels of fluoride in drinking water pose a health hazard throughout the developing world, with over 200 million people potentially impacted. In some cases, treatment methods or safer alternative sources are available, but robust, simple, affordable technologies for measuring fluoride in drinking water are absent. In this work, a simple method for fluoride detection is presented comprising a 35 nm aluminum coating on the distal tip of a length of single mode optical fiber. Broadband light is launched into the proximal end of the optical fiber and a portion of this light is reflected by the distal tip of the fiber, which is immersed in water containing an unknown concentration of dissolved fluoride. The intensity of the reflected light is detected by a photodiode connected to the proximal end of the fiber. The aluminum coating is removed from the distal tip by reaction with the dissolved fluoride at a rate that depends on the fluoride concentration and the intensity of the light reflected from the distal tip depends upon the thickness of this coating. Therefore, the rate at which the intensity of light detected by the photodiode decreases is correlated with the concentration of fluoride. The fabricated sensor measures fluoride concentration within the range of 0–5 mg L^{-1}.

1. Introduction

Elevated fluoride concentrations in drinking water present significant risk to human health [1,2]. In regions where groundwater is the main source of drinking water, most notably in the East African Rift Valley and parts of India [3]. Sri Lanka and Northern China [4], over 200 million people consume water with naturally-occurring fluoride levels that exceed the World Health Organization (WHO)-recommended limit of 1.5 mg L^{-1} [5,6]. While optimal low concentrations (0.5–1 mg L^{-1}) prevent dental caries [7]. higher concentrations (>1.5 mg L^{-1}) can lead to dental and skeletal fluorosis [8–14]. Managing the concentration of fluoride in drinking water supplies is crucial [15], and reliable, frequent measurement is essential to the effective provision of safe drinking water either by accessing alternative sources [16–20] or through affordable treatment [6,21–27].

Various methods are currently used to measure the concentration of fluoride in water [28] including ion-selective electrodes [29], mass spectrometry [30], UV–visible spectroscopy [31], and fluorescence techniques [32]. However, these methods are not suitable for field use as they require careful preparation and analysis in a laboratory setting. For communities in low-resource contexts, there is a need for a robust device that can be used in the field, such as a handheld sensor [33].

The current industry standard for field measurement of fluoride is the SPADNS method, named after the chromophoric reagent 2-(p-sulfophenylazo)-1,8-dihydroxy-3,6-napthalenedisulfonic acid) [34], developed by HACH and sold as a kit. This method measures the reaction of a red zirconium dye with fluoride in solution using a pocket colorimeter [35]. This method can measure fluoride in the range of 0.1–2 mg L^{-1} with high sensitivity [36], but its application is limited due to the requirement for manipulation of liquid reagents and possible contamination of glassware and equipment. Additionally, the SPADNS reagent for fluoride contains sodium arsenite, which is hazardous and proper disposal must follow strict protocols.

Alternative colorimetric fluoride detection methods have been developed that based on cell phone-compatible measurement kits, some of which are commercially available. In most of these methods, water is mixed with a liquid reagent in a sample chamber and characterized using the camera on a smart phone and custom software [37–39]. Using a variant of this approach, Vidan et al. react

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fluoride with the SPADNS reagent on filter paper rather than in solution, again using a cell phone camera and image processing software to quantify the analytes [40]. As with the HACH kit, all of these methods are limited by the requirement to manipulate and dispose of liquid reagents and by contamination between samples. Photographs of samples are also subject to variability in lighting and image quality.

Three studies describing fiber optic sensors for detection of fluoride in aqueous solutions have been identified in the literature; all are colorimetric in nature. Jadhav et al. measure fluoride concentration based on the interaction of the evanescent field of an in-fiber Bragg grating with a test solution to which an unspecified reagent is added. The Bragg wavelength, measured with an optical spectrum analyzer, shifts in response to changes in fluoride concentration [41]. Xiong et al. measure the concentration of fluoride using a photomultiplier tube positioned adjacent to an annular microfluidic channel, created by the space between concentric optical fiber and plastic tubing, containing the test solution mixed with a SPADNS reagent [42]. Pillai et al. measure attenuation of light transmitted through an optical fiber that is etched to enable optical interaction with the test solution to which a SPADNS reagent is added. Interrogation of this sensor is based on custom-built photo-electric circuit. All of these methods require availability of reagents or specialized optical equipment and are, therefore, not suitable for low cost field measurements.

Optical fiber sensors have been used for the detection of other chemistries, including pH monitoring [43–47], explosives detection [48,49], methane [50,51], and CO_2 and supercritical CO_2 measurement [52]. Recently, Prussian Blue has been deposited onto the tips of optical fibers and used for detection of peroxide in a fuel cell environment [53,54].

In this work, we present a simple method to detect low concentrations of fluoride in drinking water. The tip of a single mode optical fiber is coated with aluminum using sputter deposition. Immersion of the coated fiber tip in a fluoride solution then leads to removal of the coating due to the reaction between fluoride and aluminum. Removal of the coating reduces the intensity of the light that reflects from the fiber tip, as illustrated in Fig. 1. The signal associated with this intensity change is shown to be proportional to the concentration of fluoride up to 5 ppm, allowing simple, reagent-free quantitation of fluoride concentration in water at relevant concentrations for drinking water characterization.

2. Materials and methods

2.1. Chemicals

Distilled water and sodium fluoride (NaF > 0.99) (Sigma Aldrich) were used to prepare test solutions. pH was not explicitly controlled, but was consistently measured to be between 6.4 ± 0.1 for distilled water and 6.9 ± 0.1 for 80 mgL^{-1} fluoride solution. To prepare a 100 mgL^{-1} fluoride stock solution, 0.105 g of NaF was dissolved in 500 mL of distilled water. The stock solution was then diluted to prepare solutions with concentrations of 1.0 (e.g. 0.1 mL stock solution and 9.9 mL of distilled water), 2.0, 3.5, 5.0, 10.0, 20.0, 40.0, and 80.0 mgL^{-1} F- solution.

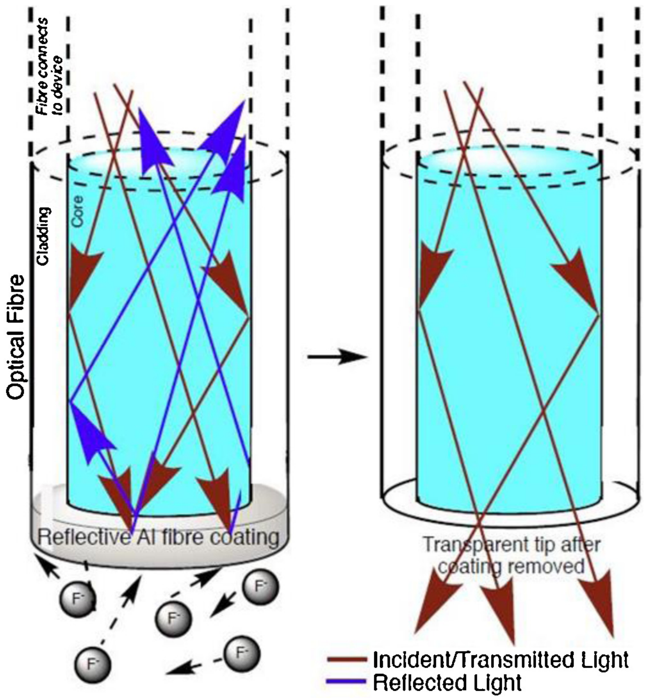


Fig. 1. The aluminum coated fiber tip. The fluoride ions remove the reflective aluminum layer from the fiber tip reducing the intensity of the reflected light.

2.2. Sensor fabrication

The outer jacket and cladding were removed from 2 cm at the distal ends of 45 lengths of single mode optical fiber (Simplex 9/125, FS.COM-China). One tip of each of these fiber lengths was sputter coated with aluminum to a thickness of 35 ± 0.5 nm (QUBE, Mantis Deposition-UK). This thickness was chosen because the sensor response time increases with coating thickness, but coatings of less than 35 nm break down easily when brought into contact with an aqueous solution.

2.3. Instrumentation

As shown in Fig. 2, the coated fiber tip is immersed in the test solution. 1550 nm light is transmitted from the broadband light source (BBS 1550, AFC Technologies), through the optical splitter

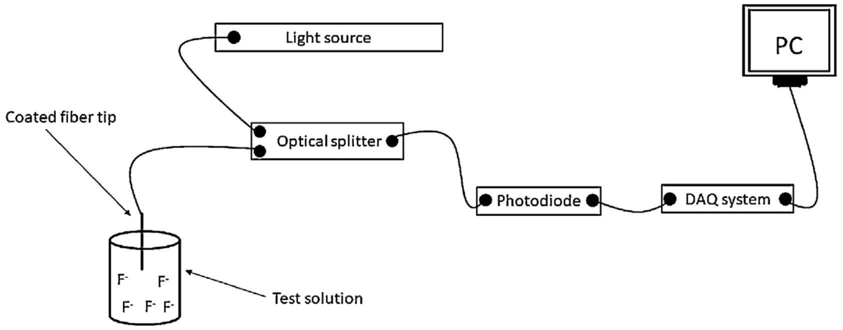
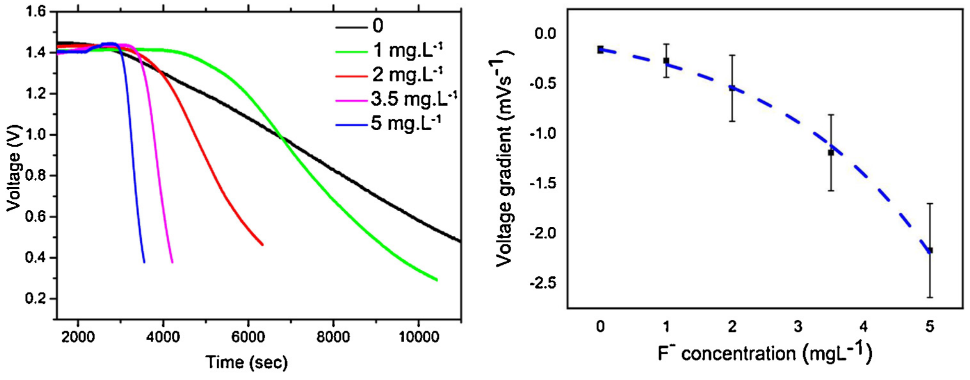


Fig. 2. Schematic drawing of the experimental set-up.



(a) (b)

Fig. 3. (a) Representative voltage gradient for single experiments with coated optical fibers immersed in 0, 1, 2, 3.5 and 5 mg L^{-1} F- solutions; (b) decline of voltage rate with the increase in concentration of F- for 0, 1, 2, 3.5 and 5 mg L^{-1} of F- (mean values for five measurements, error bars correspond to standard deviations).

(BRR-35S, Blue Road Research Inc.) and to the coated fiber tip. Light reflected from the coated tip is transmitted back through the fiber, through the optical splitter to the photodiode (FDP 510, MenloSystems) that produces voltage proportional to the intensity of the reflected light. This signal is acquired by the data acquisition (DAQ) module (NI USB-6008, National Instruments) at a rate of 10 Hz.

2.4. Test procedures

Each sensor tip was tested in a single solution sample. In total, 45 experiments were conducted using one sensor tip in each experiment. Five experiments were conducted at each of the following concentrations of F^-: 0.0, 1.0, 2.0, 3.5, 5.0, 10.0, 20.0, 40.0 and 80.0 mgL^{-1}. In each experiment, the initial signal was approximately 1.4 V and this signal was recorded until it fell below approximately 0.4 V.

3. Results and discussion

Fig. 3(a) depicts representative voltage versus time data for single experiments with coated optical fibers immersed in 0, 1.0, 2.0, 3.5 and 5.0 mgL^{-1} F- solutions as shown in Table 1. For each experiment, the voltage rate change, or *gradient*, with respect to time is calculated based on a linear fit of the data between 1.2 V and 0.6 V. These gradient values are shown in Table S1 in the Supplementary information. Fig. 3(b) depicts average voltage gradient versus F- concentration for the sensor tips at each of the solution concentrations between 0 and 5 mg L^{-1}. The gradient increases by two orders of magnitude, from -1.6×10^{-5} mVs^{-1} to -1.2×10^{-3} mVs^{-1}, over this range.

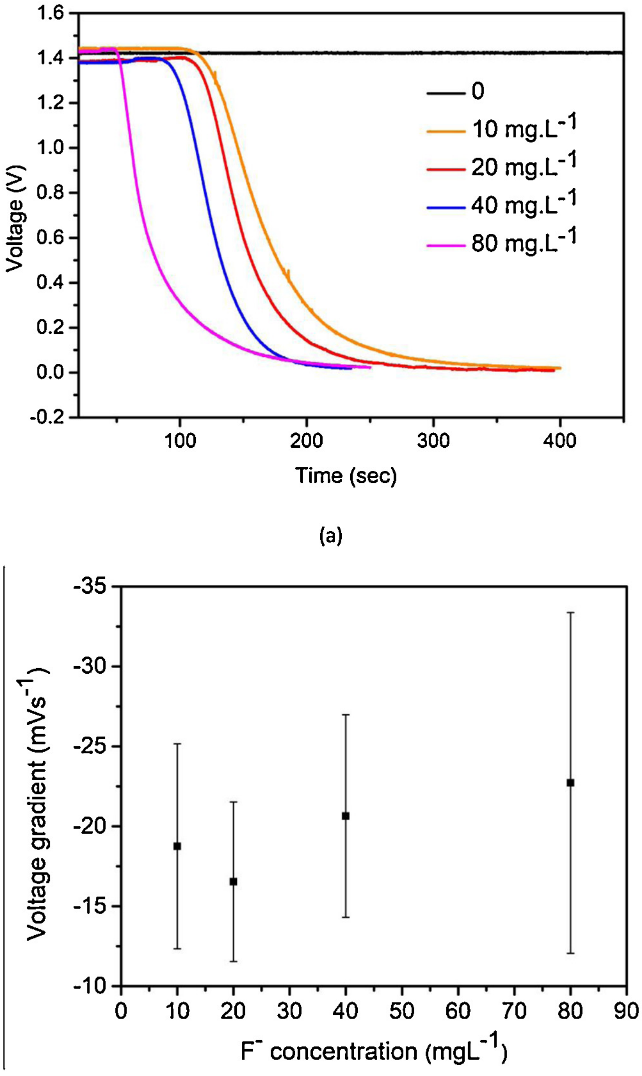
An exponential curve was fit to the 25 voltage gradient values determined from the experiments in 0, 1.0, 2.0, 3.5 and 5.0 mgL^{-1} F- solutions. The equation for this curve is:

$$\Delta V/\Delta t = (-0.297)e^{-(0.413)[F-]} \tag{1}$$

The regression coefficient for this fit is $R^2 = 0.993$. We do not postulate a specific mechanism of reaction of this complex heterogeneous system based on this fit, but rather note its empirical value for determination of the concentration of unknown samples within the 0–5 mg L^{-1} F- concentration range.

When an Al coated fiber tip is placed in contact with water, with no dissolved fluoride, amorphous oxide (Al_2O_3) forms, dissolves in water, and then precipitates as aluminum hydroxide [55]. The rate of this reaction is significantly lower than reaction of Al with fluoride (as shown in Figs. 3a and 4a)

Fig. 4(a) depicts representative voltage versus time data for single experiments with coated optical fibers immersed in 10, 20, 40,



(a)

(b)

Fig. 4. (a) characterization of the sensor using solutions with 0, 10, 20, 40, and 80 mgL^{-1} of F-; (b) decline of voltage change with the increase in concentration of F- for 10, 20, 40, and 80 mgL^{-1} of F- (mean values for five measurements, error bars correspond to standard deviations).

Table 1
Mean and standard deviation of voltage gradient based on five tests at each F^- concentration.

F^- concentration (mgL^{-1})	Mean voltage gradient ($\Delta V/\Delta t$)	Standard deviation	Linear regression coefficient (R^2)
0	-1.6×10^{-1} mVs^{-1}	$\pm 3.3 \times 10^{-2}$	0.999
1	-2.7×10^{-1} mVs^{-1}	$\pm 1.7 \times 10^{-1}$	0.997
2	-5.4×10^{-1} mVs^{-1}	$\pm 3.3 \times 10^{-1}$	0.999
3.5	-1.2 mVs^{-1}	$\pm 3.8 \times 10^{-1}$	0.999
5	-2.1 mVs^{-1}	$\pm 4.3 \times 10^{-1}$	0.999
10	-1.9×10 mVs^{-1}	± 6.4	0.999
20	-1.6×10 mVs^{-1}	± 5.0	0.999
40	-2.1×10 mVs^{-1}	± 6.3	0.999
80	-2.3×10 mVs^{-1}	± 10.7	0.994

and 80 mgL^{-1} F- solutions as shown in Table 1. For each experiment, the voltage rate change, or *gradient*, with respect to time is calculated, as described earlier. These gradient values are shown in Table S1 in the Supplementary information. Fig. 4(b) depicts average voltage gradient versus F- concentration for the sensor tips at each of the solution concentrations between 10 and 80 mgL^{-1}.

From these figures, it is clear that there is no significant difference in the rate of aluminum removal from the sensor tips as a function of F^- concentration in the 10–80 mgL^{-1} range. At high F^- concentrations, the rates of reaction are too fast to resolve by this method. Depositing a thicker aluminum layer or a metal that reacts more slowly with fluoride could improve the sensitivity of this type of sensor at high fluoride concentrations. However, towards the goal of low-cost detection of fluoride in drinking water, resolution in the 0–5 ppm concentration range, encompassing the 1.5 $mg\,L^{-1}$ WHO recommended maximum fluoride concentration limit, is of greater practical utility.

4. Conclusions

In summary, we have fabricated fluoride sensors by depositing a 35 nm thick aluminum layer on the tip of a single mode optical fiber. Exposure to an aqueous fluoride solution removes the reflective aluminum layer from the sensor tip at a rate correlated to fluoride concentration and measured by voltage change at a photodiode. The single-use optical fiber tips are inexpensively fabricated, easily coupled to a simple photodiode, and are sensitive at fluoride concentrations ranging from 0 to 5 $mg\,L^{-1}$ (encompassing the WHO maximum contaminant limit of 1.5 $mg\,L^{-1}$), making them an important first step towards the development of low-cost, robust sensors for field detection of fluoride in drinking water.

Declaration of Competing Interest

The authors declare no conflicts of interest.

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.sna.2019.111684.

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