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# Fabrication and characterization of down-tapered optical fiber pH sensor using sol-gel method

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ABSTRACT

In the present paper the fabrication and characterization of down-tapered optical fiber based pH sensor have been reported. The sensing probe is formed by fusing single mode fibers (SMF) to make it down-tapered which is spliced with multi-mode fiber (MMF). The sensing region is immobilized with sol-gel prepared by mixture of tetraethyl orthosilicate with three different pH indicators on to cladding of middle SMF. The proposed sensor utilizes intensity interrogation method and operates in low and high pH values. The maximum sensitivity of 0.49 dBm/pH is obtained with a good linear response for wide range of pH value varied from 4 to 13. In addition to high sensitivity, this sensor has additional advantages of good stability, fast response time and capable of using in remote sensing.


## 1. Introduction

The pH measurement is mostly required in many fields of sciences e.g. chemical, environmental and biomedical. Practically pH of acid and base solution is determined either by using paper strip or glass electrodes. Both types of sensors have advantages and disadvantages e.g. paper strip can give only a rough idea instead of exact numerical value while glass electrodes gives numerical value but they are expensive [1,2] and suffer poor performance with solution of low ionic strength. These drawbacks of existing pH sensors motivate researchers to look forward for the development of other alternatives of pH sensors. Amongst various kinds of available sensor technologies, optical fiber based sensors attract to scientific community due to its fascinating features. The optical fiber based sensors possess cost effective fabrication, compact in size, highly sensitive and no electromagnetic interference due to the presence of optical signal which makes fiber to use worthy in every medium.

Several types of optical fiber sensors have been already fabricated and demonstrated with various sensing utilities [3–5]. The first optical fiber pH sensor was developed by Goldstein et al. in 1980 for physiological use, which works in the range varied from 7 to 7.4 [6]. The phenol red was used as a sensing indicator and the sensor was based on the concept of color detection when come in contact of various pH solutions. After year 1980, a revolutionary modification takes place in the field of fiber based pH sensing. In this class of sensors the pH indicators are infiltrated in porous polymer via simply deposited over the sensing area of fiber [7–9]. The change in spectral characteristics takes place when the pH solution comes in contact with these coated indicators. By measuring these spectral changes we can get the corresponding pH value. In recent years, the pH sensor is fabricated and further modified by changing their structures and the deposited sensing materials [10–13].

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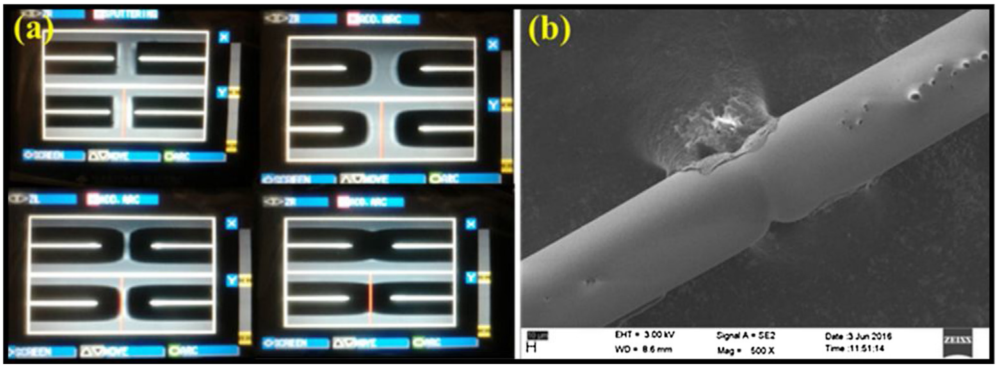


**Fig. 1.** (a) Schematic image of formation of tapered portion in SMF and (b) SEM micrograph of tapered end of fabricated probe.

**Table 1**
Shows the chemical list with their used volume.

| Solutions | Quantity |
|---|---|
| TEOS | 20 ml |
| Anhydrous ethanol | 20 ml |
| Deionized water | 1 ml |
| Cresol red | 26 mg |
| Chlorophenol red | 26 mg |
| Bromothymol blue | 36 mg |

Here, a cost effective pH sensor based on excitation of cladding modes in SMF is presented. The probe has been prepared by immobilizing the indicators on the cladding after activating $OH^-$ group on it. In sol-gel preparation the mixture of three pH indicators are used to obtain the wide pH range [14,15]. The leakage of launched light from the tapered portion causes the excitation of cladding modes of SMF which interact with the analyte via deposited sol-gel. The changes in the power with respect to the changes in pH are recorded by power meter. Investigations have been also carried out at different stirring time to study the variation in sensitivity with viscosity of sol-gel. In previously reported papers the sensors are required large probe size, chemical tapering and surface modification for better sensitivity [10,12,16] while our fabricated sensor requires simple splicing and sol-gel deposition to achieve high sensitivity.

## 2. Probe fabrication

In fabricated probe, SMF and MMF of core/cladding diameter of 8/125 and 50/125 respectively were purchased from Thor lab. The sensing probe was fabricated by fusion splicing method under optimized conditions as our previously published article [17]. The plastic jacket was removed from 30 mm length of SMF and sensing matrix was deposited onto removed jacket portion. The tapering process was done by using fusion splicer SUMITOMO T-39. Our sensor consists of a down-tapered SMF at one end and stubbing of MMF at another end. Firstly, as shown in Fig. 1(a), two cleaned SMF was placed in the splicer under optimized arc discharge to form ellipsoid heads and then fused together by giving further arc discharges to fabricate down-tapered sensing head. Fig. 1(b) shows the microscopic image of the down-tapered portion fabricated in SMF.

## 3. Synthesis of sensing matrix and experimental setup

All materials used in the synthesis of sol-gel are purchased from Sigma-Aldrich. Sol-gel method is widely used in the formation of chemosensors that requires low temperature to be produced. This method contains a complex reaction that involves mixture of various solvent along with some metal alkoxide at low temperature to prepare a homogenous solution [18,19]. The chemical which is used in the sol-gel preparation are listed below in Table 1 as per there used volume.

The sol-gel was prepared by mixing of 20 ml TEOS, followed by 20 ml of anhydrous ethanol, 1 ml of deionized water along with 26 mg of cresol red and chlorophenol red respectively, with 36 mg of bromothymol blue. The mixture was then stirred at constant temperature 60 °C before being used. TEOS was used as a precursor which gives the porous silica glass of less refractive index in comparison to the cladding to maintain the waveguide condition. Here we use three different pH indicators due to their different pH ranges and hence their simultaneous mixing enhances the range and covered a broad pH value [14,20,21]. Before deposition of sol-gel, the OH group was activated over the cladding region of SMF by treating it with 69% $HNO_3$ for 5 min.

To deposit prepared sol-gel on the cladded portion, dip coating method was used here out of various coating methods. The speed of dip coater was fixed at 70 mm/min with the two minutes of wet and four minutes of dry time. When the cladded portion is immersed and pulled in the prepared matrix, the activated OH group form bonds with the silica molecule and