A Comparative Study of Fiber Optic Humidity Sensors Based on Chitosan and Agarose

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Abstract: A comparative study of two biopolymer based fiber optic humidity sensors is presented in this paper. Sensing elements Agarose and Chitosan swells in the presence of water vapour and undergoes changes in refractive index and modulates the intensity of light propagating through a fiber with Agarose or Chitosan as cladding. Copyright © 2007 IFSA.

Keywords: Fiber optic sensor, Humidity sensor, Swelling polymer, Chitosan, Agarose

1. Introduction

Measurement of humidity is required in a range of numerous applications, including the meteorological services, the chemical and food processing industry, civil engineering, air-conditioning, horticulture, and electronics processing. Therefore, development of a low-cost humidity sensor with fast response assumes great importance. Optical fiber humidity sensors offer specific advantages, such as small size and weight, immunity to electromagnetic interference, noncorrosiveness and remote operation in comparison with their conventional electronic counterparts. Hence, a wide range of optical fiber humidity sensors have been reported in the literature [1-16]. Most of the fiber optic humidity sensors work on the basis of intensity modulation in which the light propagating through the optical fiber is absorbed or scattered as the humidity changes [6-10]. Most common method is based on the absorption/scattering of the “evanescent tail” of the light passing through the optical fiber [7-10]. Another method is based on the swelling nature of hygroscopic materials which causes refractive index changes in accordance with the humidity and modulates the light propagating through the fiber [11-16]. The latter phenomenon has been employed in the design and development of simple,
low cost and reliable fiber optic humidity sensors presented in this paper. The swelling polymers used in our work are Chitosan and Agarose. To the best of our knowledge, this is the first time that Chitosan have been used in developing a humidity sensor.

2. Agarose and Chitosan Basics

Chitosan is a fiber-like substance derived from Chitin. Principal source of Chitin is shellfish waste such as shrimps, crabs, and crawfish. Deacetylation of Chitin gives Chitosan. It is soluble in dilute acidic solutions below pH 6.0 [17, 18]. Thin films fabricated from solutions of chitosan and acetic acid have refractive indices approximately 1.45 and there is nearly no absorption in the range of 300 to 2700 nm. Agarose is an unbranched polysaccharide obtained from the cell walls of some species of red algae or seaweed. Chemically, Agarose is a polymer made up of subunits of the sugar galactose. It is soluble in hot (>35°C) water [15]. The property of thin film formation, water binding capacity and refractive index variation on water adsorption of these biopolymers [15, 19] can be exploited for the development of fiber optic humidity sensors.

3. Theory

The materials used as the functional cladding layer in the sensing region are Chitosan and Agarose, which swells in the presence of water without dissolving at room temperature. The variation in the refractive index \( n \) of these swelling polymers with respect to humidity \( H \) is obtained from Lorenz-Lorentz relation as [20],

\[
\frac{dn}{dH} = \frac{(n^2 + 2)^2}{6n} k_m S (1 - \frac{f}{f_c}),
\]

(1)

where \( k_m \) is the molar refraction divided by the molecular weight of water, \( S \) is the moisture solubility of the polymer, parameter \( f \) (0<\( f \)<1) is the fraction of the absorbed moisture that contributes to an increase in polymer volume, and is expected to depend on temperature, In Eq. (1), critical value \( f_c \) defined by

\[
f_c = k_m \rho_m \frac{n_p^2 + 2}{n_p^2 - 1},
\]

where \( n_p \) is the refractive index of the polymer without any moisture and \( \rho_m \) density of water.

The output power \( P_{out} \) of the optical fiber sensor head with respect to the refractive index \( n \) of sensing layer is given by [21]:

\[
P_{out} = P_{in} \left( \frac{n_1^2 - n^2}{n_1^2 - n_2^2} \right),
\]

(2)

where \( n_1 \) is the refractive index of the core, \( n_2 \) is the refractive index of the cladding, \( P_{in} \) represents the total power injected into the guided modes of the fibre from the source.

In the dry state the refractive index of the Chitosan/Agarose cladding layer is larger than that of the fiber cladding and it operates in the leaky mode for higher order modes. In the humid air Chitosan/Agarose swells, its refractive index decreases and most of the higher order modes are guided and the intensity of transmitted light increases. Thus relative humidity can be determined by measuring
the output intensity of light passing through the sensing region. Fig. 1 schematically shows the leaky and guided modes in the sensor head.

![Swelling Polymer Chitosan/Agarose](image)

**Fig. 1.** Leaky and guided modes of the sensor head.

On the basis of the above-mentioned principle, the output light intensity passing through the sensor head can be calculated theoretically for our sensor head using a ray tracing method [13]. Output power of the sensor head is represented mathematically by equation (3),

\[
P_{\text{out}} = \sum_{\theta=0}^{\theta_{cs}} P(\theta) + \sum_{\theta=\theta_{cs}} P(\theta)r^{m},
\]

where \(\theta_c\) is the critical angle in the input-side of the plastic cladded silica (PCS) fiber, \(r(\theta)\) is the reflection coefficient, \(m\) is the reflection number for the sensor head and \(\theta_{cs}\) is the critical angle in the sensor head.

### 4. Sensor Element Fabrication

The sensor element fabrication includes fiber preparation and deposition of the Agarose/Chitosan film on the prepared fiber. The fiber used for the sensor element fabrication is a multimode, step index, plastic cladded silica fiber of core diameter 400 \(\mu\)m with a numerical aperture of 0.37 (Type F-MBC). 5 cm length of the cladding is removed from the middle portion of the fiber. The ends of the fiber are polished well. The Chitosan sensor head is fabricated by dip coating the uncladded portion of a fiber in a Chitosan solution prepared by dissolving 1 % Chitosan in 4 % acetic acid solution. The Agarose sensor head is fabricated by coating the uncladded portion of a fiber with hot (>35 °C) 1 % Agarose solution [15]. The coated fibers are kept for one day at room temperature until it is partially dehydrated or reaches the equilibrium with the environment.

### 5. Experimental Setup

The experimental setup (Fig. 2) consists of an LED, detector with signal analysis and the humidity chamber in which the Chitosan/Agarose coated fiber is fixed. Source used for the experiment is a red LED emitting at 636 nm. A low power silicon photo detector (Newport 818-IR) was used with a power meter (Newport make, Model 1815C) for recording the output. Humidity chamber is made of...
borosilicate glass. Two fiber holders are fixed at the two side walls of the chamber. Humidity measuring unit used for calibration has a measuring range of 10 % - 95 % RH. The sensor head of this humidity measuring equipment is inserted into the chamber.

Fig. 2. Experimental setup.

The aerator is used to pump air into humidity chamber via ethylene glycol (dry air) or water (humid air). Air bubbled through ethylene glycol will bring down the chamber humidity to 20 % RH, Also, passing nitrogen gas into the chamber can bring down the chamber humidity to 17 % RH. The whole setup can be made compact if the sensor head used for calibration is small.

6. Results and Discussions

Using both Chitosan and Agarose coated sensor head, power outputs were taken for different humidity values and were plotted with humidity on the X-axis and normalized power on the Y-axis. The normalized power is obtained as

\[ P_N (\text{dB}) = 10 \log \left( \frac{P_o/p \text{ at a value of Humidity}}{P_o/p \text{ at max value of Humidity}} \right) \]  

The results are plotted in Fig. 3 & Fig. 4 for Agarose sensor head and Chitosan sensor head respectively and it is observed from the figures that the normalized output power varies almost linearly with humidity for these biopolymer coated fiber optic sensor heads. The above graphs also establish the reversibility of the fiber optic humidity sensors. Long term stability of the sensor head is studied by taking the humidity response for different days (data not shown) and it is observed that the response is consistent within the accuracy limit of +/-5% for a humidity variation of 17-95 % RH for Chitosan coated sensor head and for Agarose coated sensor head it is stable within the accuracy limit of +/-1% for a humidity variation of 40-95 % RH.
To measure the refractive index of the Chitosan film, film of thickness 15 µm is made from the Chitosan-acetic acid solution. The measured refractive index using Brewster angle method is approximately 1.45. The absorption spectrum of the film is taken using the UV/VIS/NIR Spectrophotometer (Make Jasco, Model V-570). It is observed that there is nearly no absorption in the range of 300 to 2000 nm.

Investigations were also carried out by changing the wavelength of the LED and by changing the probe length. In both these cases no appreciable variation in the sensitivity has been observed. Further,
investigations have been carried out on the temporal response of the sensors using LabVIEW software and it is observed that the sensor showed fast response to humidity changes. A comparison of the performance of Chitosan coated sensor head and Agarose coated sensor head is given in Table 1.

<table>
<thead>
<tr>
<th></th>
<th>Chitosan coated sensor head</th>
<th>Agarose coated sensor head</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Sensitivity</strong></td>
<td>0.001 dB/RH</td>
<td>0.001 dB/RH</td>
</tr>
<tr>
<td><strong>Response time</strong></td>
<td>2 seconds</td>
<td>3 seconds</td>
</tr>
<tr>
<td><strong>Linear response</strong></td>
<td>17-95 %RH (accuracy +/-5%)</td>
<td>40-95 %RH (accuracy +/-1%)</td>
</tr>
</tbody>
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Employment of these humidity sensors could lead to improvement in health care, since similar response time of commercial humidity sensors is of the order of several seconds. Fig. 5 demonstrates the ability of our humidity sensor based on Chitosan for on-line monitoring of breathing. This clearly establishes the sensitivity and fast response of the device to small variations in humidity. Also its simplicity and relative low cost make it an interesting possibility for humidity sensing.

![Continuous breathing](image)

**Fig. 5.** Response of the sensor to continuous breathing.

7. Conclusions

Simple fiber optic humidity sensors using swelling polymers as humidity sensing materials have been studied. The materials Chitosan and Agarose deposited as the cladding of an optical fiber swells in the presence of water molecules and are demonstrated to be suitable in humidity sensor applications. These sensors are reversible in nature. Stability of these sensors is also very good. In addition, as this sensor has a fast response time, it could be used for real-time humidity monitoring and for applications as a breathing condition monitor. This study can also be useful for the fabrication of gas sensors.
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