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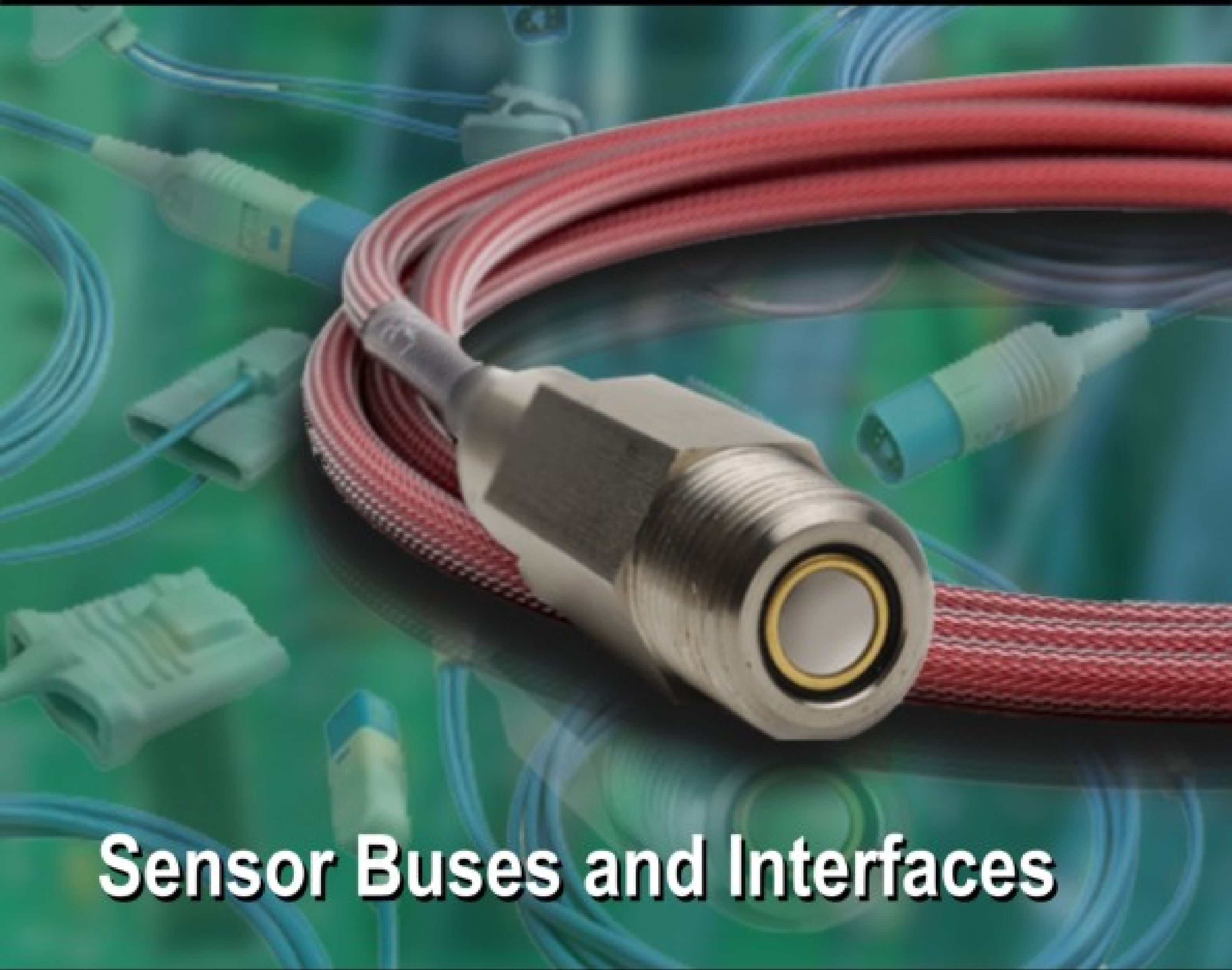

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Optical Fiber Humidity Sensor Based on Ag Nanoparticles Dispersed in Leaf Extract of Alstonia Scholaris

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Abstract: An optical fiber with a clad of Ag nanoparticles dispersed in leaf extract of Alstonia Scholaris is used as an optical humidity sensor. The fabricated sensor showed response to humidity in the range of 40-95%. The specialty of this sensor is that it can be used when stored at room temperature (25 ^{o}C) up to a maximum of 25 days with 90% retention of original sensitivity. These humidity sensing bio-films showed good operational efficiency for 5 cycles. The plastic optical fiber is versatile and can be used easily for humidity measurement with high sensitivity. The sensor exhibited a short response time of 4-5 sec. and recovery time of 45 sec with repeatability, reproducibility and low hysteresis effect. This Ag dispersed in leaf extract of Alstonia Scholaris showed higher humidity response compared to response shown by the leaf extract alone. *Copyright © 2008 IFSA.*

Keywords: Humidity biosensor, Optical waveguide, Relative humidity

1. Introduction

The need for simple, rapid, and continuous in-situ monitoring techniques in a broad range of areas, e.g. medical, pharmaceutical, environmental, defense, bioprocessing, or food technology has lead to the development of biosensor. Biological systems such as tissues, microorganisms, enzymes, antibodies, nucleic acids, etc. are combined with a physico-chemical transducer which may be an optical, electrochemical, thermoelectric, piezoelectric, etc.[1] Many optical methods that are promising for chemical sensing have been reported. In general, conventional optical and spectrometric instruments are built on a large scale. In recent years, a compact-sized chemical sensor composed of micro optical elements, such as optical fibers, waveguides and microprisms, has been developed for measuring chemical species [2].

Recently various kinds of optical waveguides have been used in integrated opto-chemical [3-7] and biosensors [8-11]. Robustness, potential for integration with other optoelectronic components for compactness, and relative ease in applying specific coatings on the waveguide surface for evanescent wave sensing are the advantages of waveguide sensors. The biochemical sensitivity is obtained by choosing a proper biomaterial as clad which may be immobilized in a suitable matrix. The biomaterial can be cladded often much easily and by different processes on the planar surface of guide, where the clad acts as an interacting region to control the evanescent mode losses. The cladded optical waveguide with high sensitivity is the most attractive transducer for chemical or physical analysis, dependent on transmitted light intensity as well as polarization. The electric field associated with the light wave propagating in the waveguide layer is sufficiently strong on the surface of the optical waveguide which is an interesting feature. Hence highly sensitive optical monitoring can be performed for chemical species located at the optical waveguide surface on the basis of absorption and scattering of light [12, 13].

Measurement of humidity is required in numerous applications including the meteorological services, chemical and food industry, civil engineering, air conditioning, agriculture and electronic processing. Humidity sensors are of increasing interest in electronic control systems [14-23]. Therefore the relative humidity measurements have been extensively studied and a remarkable progress has been made [22]. Based on the two properties i.e. electrical and optical, different humidity sensors are studied. Z. A. Ansari et al. [14] has reported gas and humidity sensors using planar optical waveguide with screen printed thick films of semiconducting oxides. A highly sensitive optical humidity probe based on reflectance measurements has been made using Nafion(R) - crystal violet (CV) films. This sensor can be used to calibrate relative humidity in the range of 0-0.25% with a detection limit of 0.018% RH and exhibits low hysteresis. [24] An evanescent-wave optical fibre humidity sensor using $CoCl_2$ doped thin polymer film coated on the bare fibre core is reported by Sunil K. et al [23].A plastic optical fibre humidity sensor using hydroxyethylcellulose is reported by Muto et al [25]. Starch iodine films respond to water vapour as reported by Skrdla et al. [26]. A hydrophilic agarose gel optical fibre humidity sensor in the range of 30-80% was fabricated using by Bariàin et al [27]. Very few biomaterials are reported for humidity sensing in literature.

Recently various kinds of optical waveguides have been introduced in integrated opto-chemical and biosensors, in which the light absorption or the refractive index changes due to analytes on sensor surface is monitored as indicated above. Following this idea the paper presents use Ag nanoparticles incorporated in a biomaterial as a sensitive clad on the optical waveguide surface for sensing relative humidity.

2. Experimentation

2.1. Sensor Fabrication and Characterization

Leaves were picked from Alstonia Scholaris plant (Saptaparni), and were placed in cold tap water and washed thoroughly. The turgid leaves were torn by hand and ground to a smooth paste using a mortar pestle. The extract was removed and then filtered. The extract, so formed was then used for further experiments. The chlorophyll content is calculated using the standard formulae [28].

To fabricate the sensor, plastic optical fibre is used. The cladding of the plastic optical fibre having 1mm diameter and a length of 30 cm is removed from the central portion of the fibre, where the humidity sensitive film is then carefully dip coated with the cladding material. The fiber used for sensing was bent to a 'U' shape with a bent radius of 7 mm taking into considerations for the penetration depth as reported by Gupta et al. [29, 30] and Sunil et al [23]. The plastic optical fibre has a polymethylmethacrylate (PMMA) core of approximately 980 μm thickness, with 20 μm thick cladding made of fluoride containing carbon polymer. The refractive indices of the core and cladding are 1.492 and 1.417 respectively. The total diameter of the plastic optical fibre is 2.2 mm with PVC protecting sheath. The ends of the plastic optical fibre are connected to a transmitter Siemens and receiver.

PFC (Plastic Fibre Components) low cost emitter- SFH 450 having a peak emission wavelength of 950 nm and a detector-phototransistor SFH350 (responsivity of 0.3 micro-amp/micro-watt) are used. The advantage of the Siemens PFC's is the housing aperture into which a plastic optical fibre is introduced without having to remove the cladding, with an additional benefit of directly centering the fibre onto the transmitter and a detector. The transmitter and the detector is powered by +5V supply. The experimental setup is realized as shown in Fig. 1. A dome (1.6 liter capacity) is used for creating required RH. RH values are adjusted by proper inlet of wet/dry air at room temperature (30 ºC). The transmitted output light intensity is measured with respect to relative humidity. The change in the RH% is measured directly, using a precalibrated hygrometer (Vaisala). The output voltage of the receiver (detector) as a result of change in humidity is measured using a Rishcom digital multimeter.

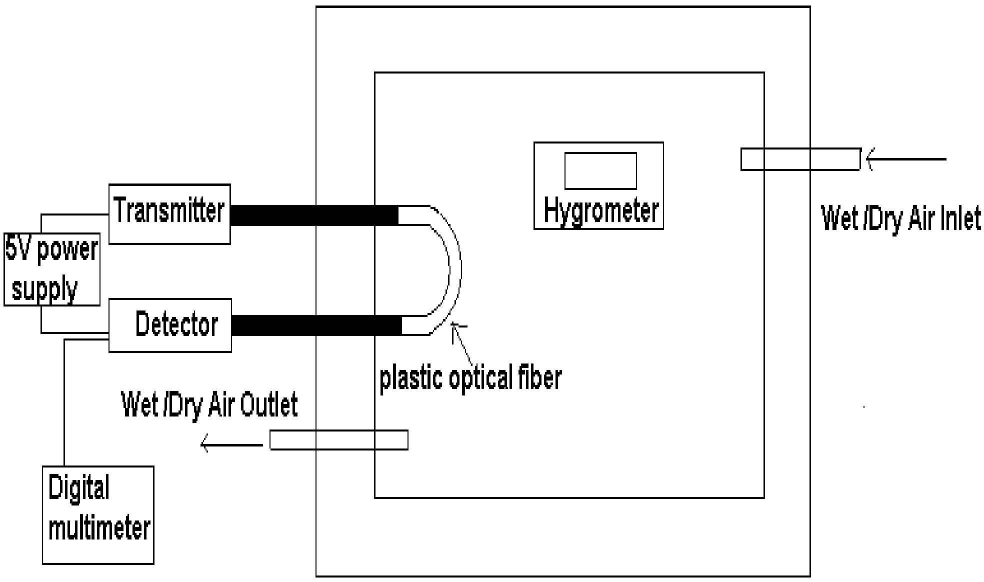

Fig. 1. Experimental setup of the sensor characterization system.

First the length of cladding was optimized and then the concentration dependence was studied. Then, the Ag nanoparticles, synthesized by electrochemical method [31] were added in different percentages to study the enhancement of sensitivity. The change in the RH% is measured directly, using a precalibrated hygrometer (Vaisala Humidity & Temperature Indicator HMI 31). The output voltage of the receiver (detector) as a result of change in humidity is measured using a Rishcom digital multimeter. The material characterization was done by SEM, and UV-Vis.

The sensitivity is defined as the change in the transmitted output (mV) per unit change (mV) per unit change in RH% i.e. δ (mV)/ δ (RH %). The response time is measured for the RH transition from low level to high level i.e. to air ambient. The converse is done for measuring the recovery time. The maximum difference in two outputs i.e. increasing and decreasing cycles of RH, at constant RH level is defined as hysteresis.

3. Results and Discussions

The SEM micrographs are as seen in Fig. 2. A mesh work like structure with well spread granular molecules on the surface is seen. For lower chlorophyll content, the uneven pores are reducing and molecules are well spread providing more sites for interaction. There is an increase in the pore size. Clusters of biomolecules are seen to be uniformly distributed within the clad. The well spread molecules may be providing more area for interaction. Hence, the humidity response is better for lower chlorophyll content film.

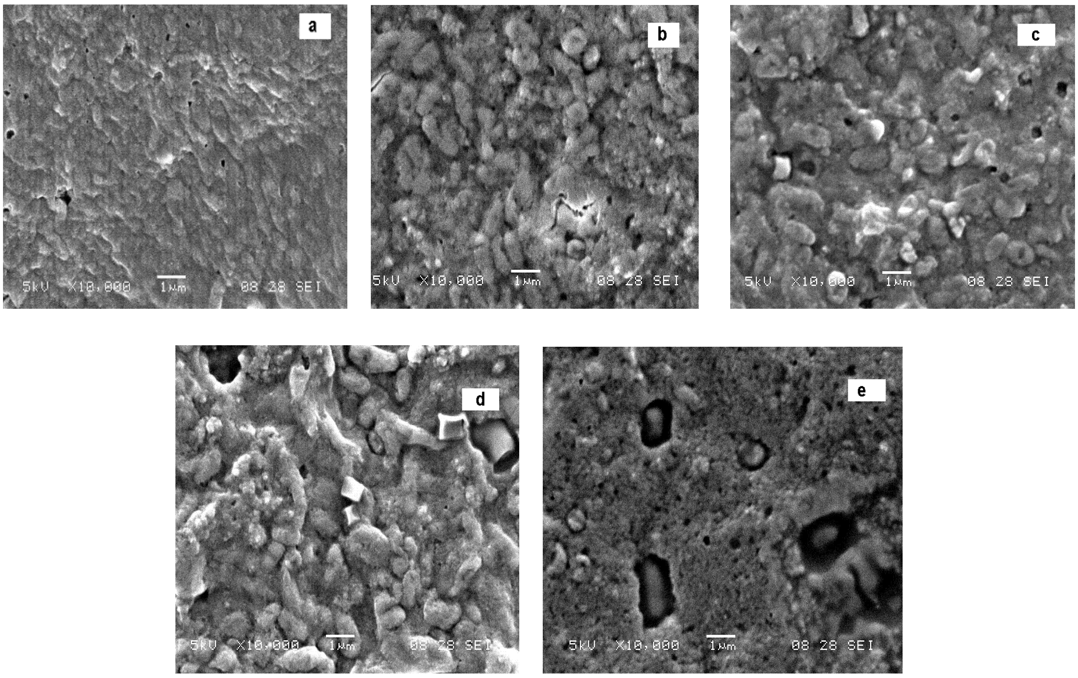

Fig. 2. Scanning electron micrographs of films with decreasing chlorophyll content (i.e. 0.386, 0.227, 0.218, 0.134 and 0.110. mgs.chl/cc).

The UV –Vis spectrum (Figs. 3 and 4) shows absorption peaks at 435 nm, 475 nm, 650 nm and 675 nm. Gross et al have reported the approximate absorbance maxima of 430 and 662 nm for Chlorophyll a and chlorophyll b the approximate absorbance maxima of 453 and 642 nm. The exact positions may vary with solvents. Here, acetone is used as a solvent. The peaks at 435 and 675 nm perhaps represent the presence of chlorophyll a and at 475 and 650 nm are representing the presence of chlorophyll b [32]. In an organic solution, chlorophyll has two absorption maxima in the visible spectrum with wavelengths shorter than 720 nm. The short-lived fluorescent state is produced by the excitation of either the blue or red absorption band. Electrons return to their ground states by emission of fluorescence, dissipation of heat or by a chemical reaction. A possibility of transferring the energy from one pigment molecule to another also exists. This is referred to as a homogeneous energy migration, and it results in a depolarization and a quenching of the fluorescence. An excited chlorophyll molecule transfers one electron to an acceptor molecule in the adjacent lipid layer and recovers it from the protein layer. With the addition of Ag nanoparticles, a new absorption peak is obtained at 400 nm.

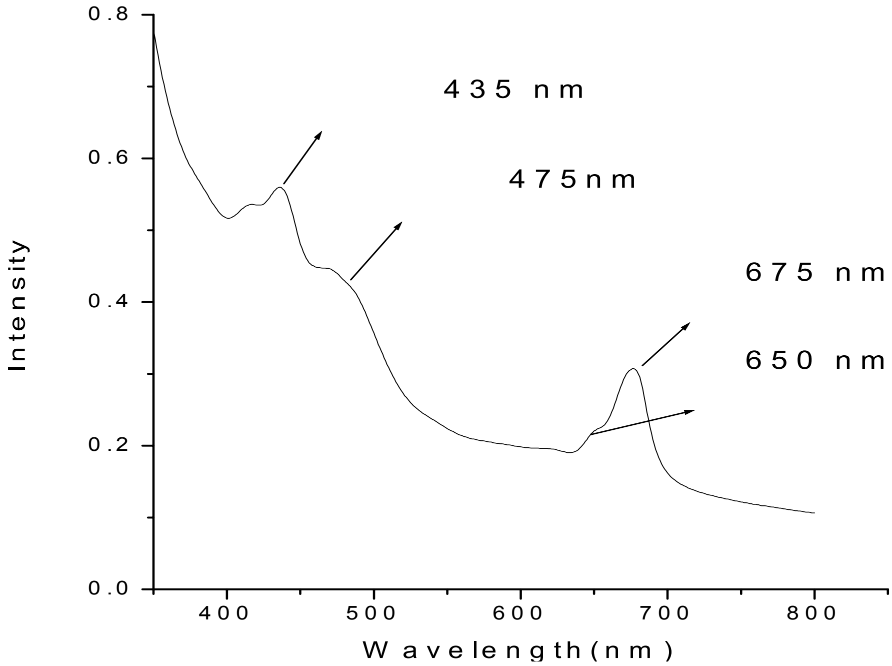

Fig. 3. UV-vis absorption spectra of extract in acetone medium.

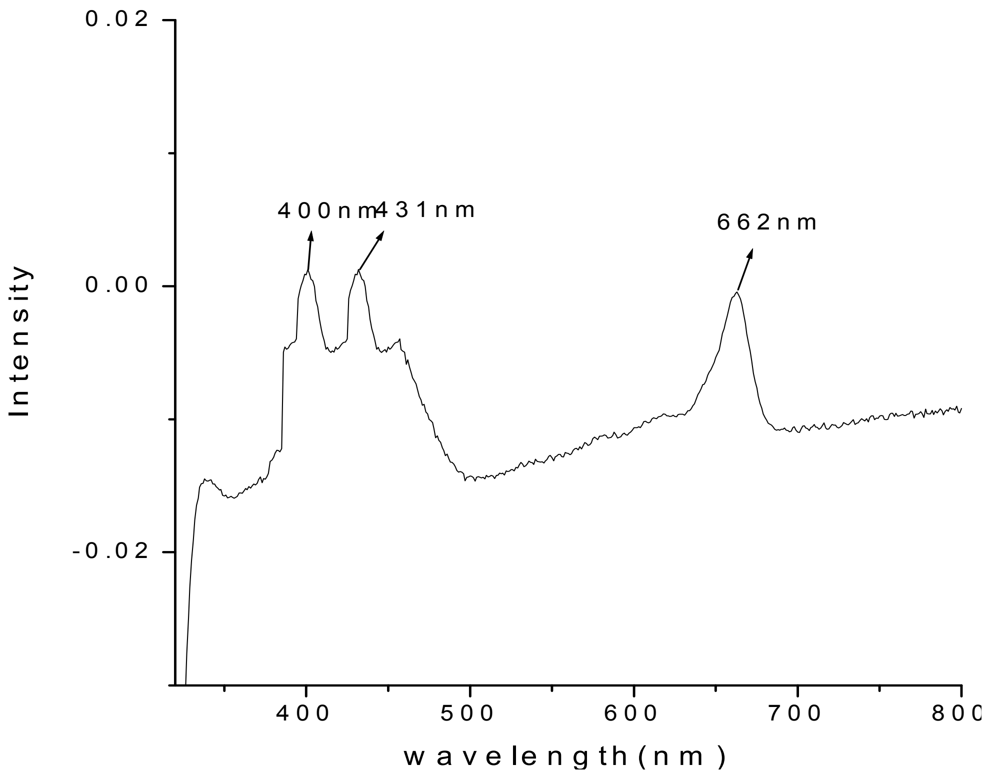

Fig. 4. UV-vis absorption spectra of extract with Ag particles in acetone medium.

A small portion of the optical power in the guided modes, extended to the cladding region, interacts with the coated sensing biofilm is based on the phenomenon of the evanescent wave adsorption. The guide has a refractive index of 1.53 which is higher than the refractive index of the coated film used; 1.484 for a clad of chlorophyll content of 0.110 mgs.chl/cc, and it decreases to lower values with increasing chlorophyll content. This results in leaky or radiation field in the clad that maintains the total internal reflection in the cladding. Confining more and more power in the optical guide. With increase in the humidity, more light is confined in the guide. Thus, output voltage increases with the increase in humidity as observed in Figs. 5 and 6; effectively increasing the sensitivity.

Fig. 5 shows the humidity response for variation in the length of the coated bio clad. (Length -10 mm, 9 mm, 7 mm, 5 mm). As per these variations, 9 mm clad length showing maximum sensitivity is considered as an 'optimal length'. The increase in sensitivity with the length of clad may be due to the availability of large interacting sites. This causes larger amount of hydroxyl ion adsorption by the clad enhancing the changes in optical properties giving rise to higher sensitivity. The overall sensitivity of the sensor increases upto 9mm clad length giving maximum confinement to the transmitted laser beam and later on decreasing giving low response with increasing length. At higher lengths the cladding material might be absorbing the transmitted light in addition to the obvious losses offered by the guide which decreases the intensity.

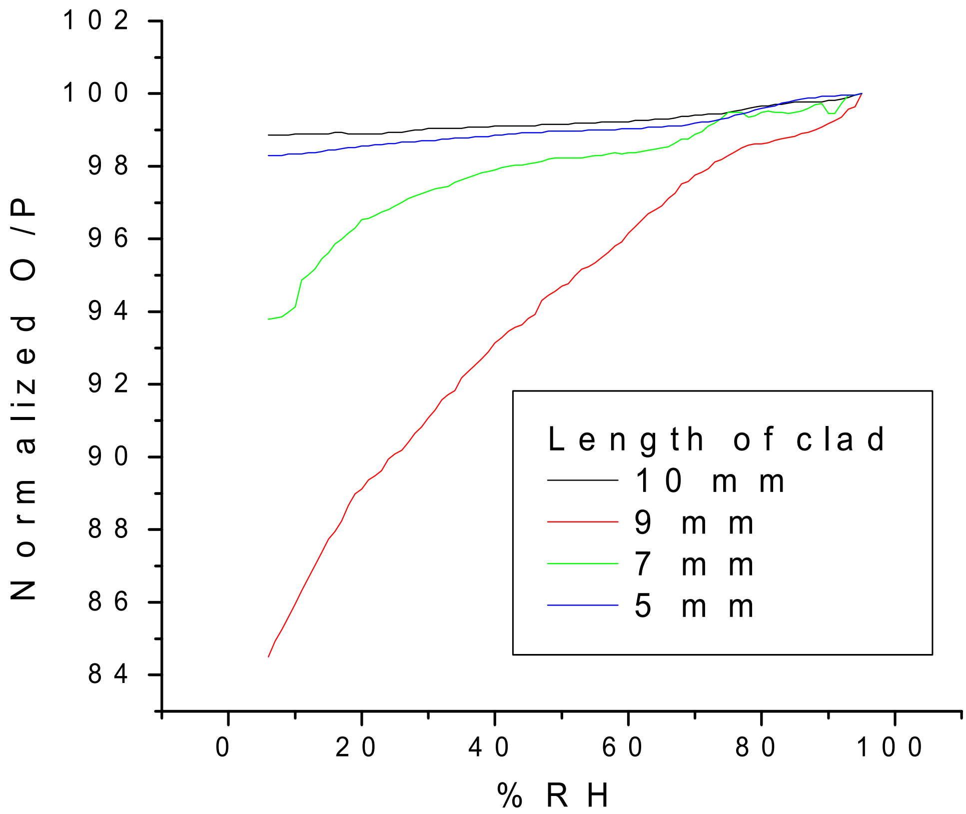

Fig. 5. Humidity sensing with variation of clad length.

Experimentation for finding the optimum chlorophyll concentration of the clad for maximum sensitivity for 9 mm clad is worked out. The chlorophyll concentration of the biofilms was varied from 0.386 mgs.chl/cc to about 0.110 mgs.chl/cc. As the relative humidity increases, the sensitivity decreases for increasing chlorophyll content as shown in Fig. 6. Chlorophyll content of 0.110 mgs.chl /cc shows a maximum sensitivity.

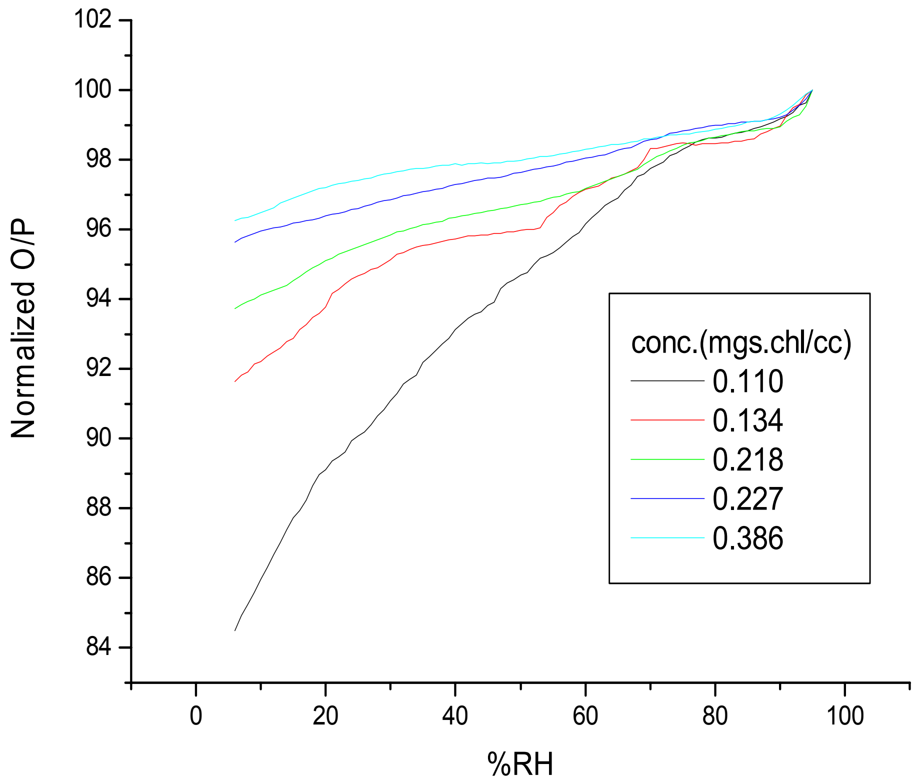

Fig. 6. Humidity sensing for clads with different chlorophyll content.

From the SEM image (Fig. 2) of film with lower chlorophyll content, it is evident that the clad is more porous, hence offers more sites for interaction of water molecules offering maximum sensitivity, whereas for higher concentrations voids are less in numbers giving lower sensitivity for humidity. The sensitivity curve shows different slopes, giving three different humidity regions. Fig. 7 gives the response of bio-clad containing different percentages of Ag nanoparticles to humidity.

The refractive index of the coated film of Ag nanoparticles incorporated in Bio-extract used is lesser than the waveguide refractive index of 1.53. The clad containing 100% Ag nanoparticle content has a refractive index of 1.50 and decreases to lower values with increasing percentages of Ag content as given in the Table 1 as measured by Abele's method, resulting in more leaky or radiation field in the cladding which maintains the total internal reflection in the cladding. This confines more and more power in the optical fibre. With increase in the humidity, the refractive index of the biomaterial further decreases and continues to decrease further with increase in humidity of the chamber, confining more light in the fibre. Thus, output voltage increases with the increase in humidity; effectively increasing the sensitivity, as observed in Fig. 7.

The sensing occurs due to the adsorption of water molecules on the surface of the film. The dissociation of water molecule can be considered as surface reaction leading to the formation of surface oxides. This effectively changes the boundary conditions at clad-guide interface increasing the beam confinement in the guide. Therefore, the output intensity through the cladded guide increases. The three regions in sensitivity curves indicate different dominant phenomena in the process. At a low humidity (region I), when adsorption starts on the surface, a layer of hydroxyl groups is formed. The water molecules are chemisorbed through a dissociative mechanism by which two hydroxyls per water molecule are formed. Thus, in the first region as the humidity decreases output intensity decreases but sensitivity in this region is higher because RI of the film decreases. In intermediate RH range, monolayers of water molecules will be adsorbed along sites available on the film. Here, light transmitted through the fibre gets more confined than in the lower humidity region. At higher RH, (region III) water adsorption by the cladding becomes multilayer adsorption in the pores of the clad penetrating deeper up to the clad guide interface. The refractive index of the clad further decreases resulting in more confinement of light in the fibre and hence maximum output is transmitted.

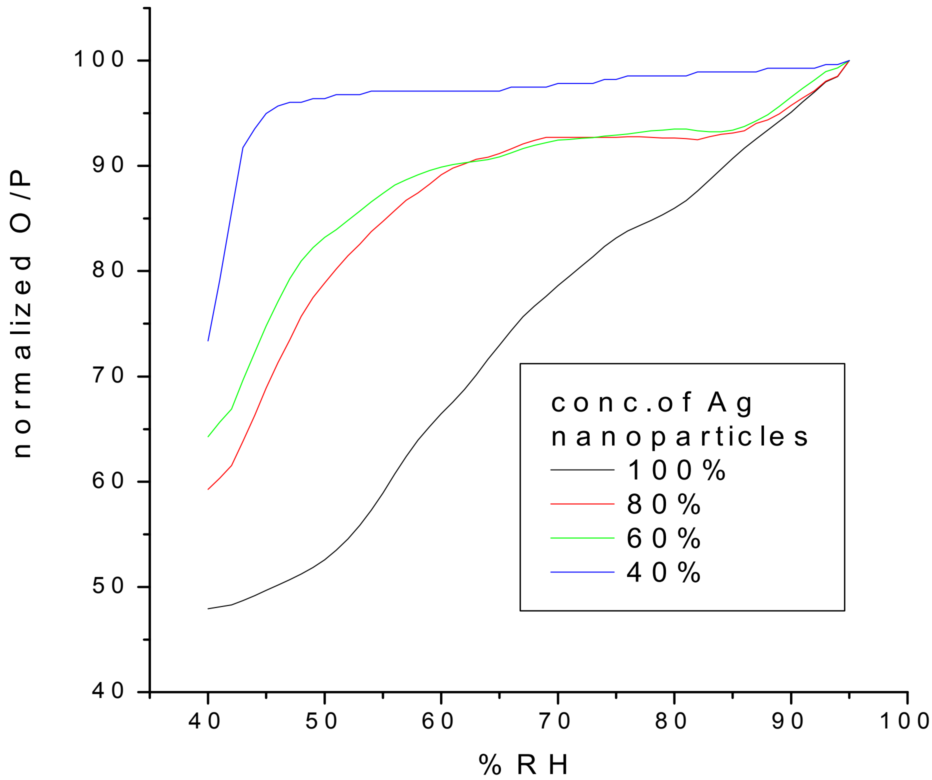

Fig. 7. Response of bio-clad containing different percentages of Ag nanoparticles to humidity.

Table 1. Region wise sensitivity for different percentages of Ag nanoparticles dispersed in extract.

Concentration of Ag nanoparticles	Refractive index	Region 1(V/RH)	Region 2(V/RH)	Region 3(V/RH)
100%	1.50	1.719(40-53%RH)	0.539(53-78%RH)	0.905(78-95%RH)
80%	1.485	1.695(40-58%RH)	0.200(58-82%RH)	0.669(82-95%RH)
60%	1.435	1.201(40-55%RH)	0.190(55-84%RH)	0.562(84-95%RH)
40%	1.429	0.654(40-48%RH)	0.054(48-65%RH)	0.080(65-95%RH)

The humidity range is enhanced from 70% to 40% for Ag dispersed in extract. Comparing the sensitivities of leaf extract and Ag dispersed leaf extract clad the later shows higher sensitivity.

Hysteresis, (Fig. 8) defined as the maximum difference in the two outputs (increasing and decreasing RH cycle) at the same RH level, is observed to be nearly 2%.

The repeatability (Fig. 9) and reproducibility (Fig. 10) of the sensor was found to be very encouraging. About 3% uncertainty from cycle to cycle and uncertainty of about 4% from sample to sample indicate better repeatability and reproducibility. The sensor shows quick response of 4-5 seconds (40 to 95% RH) and recovery time of 45 sec (95 to 40 % RH).

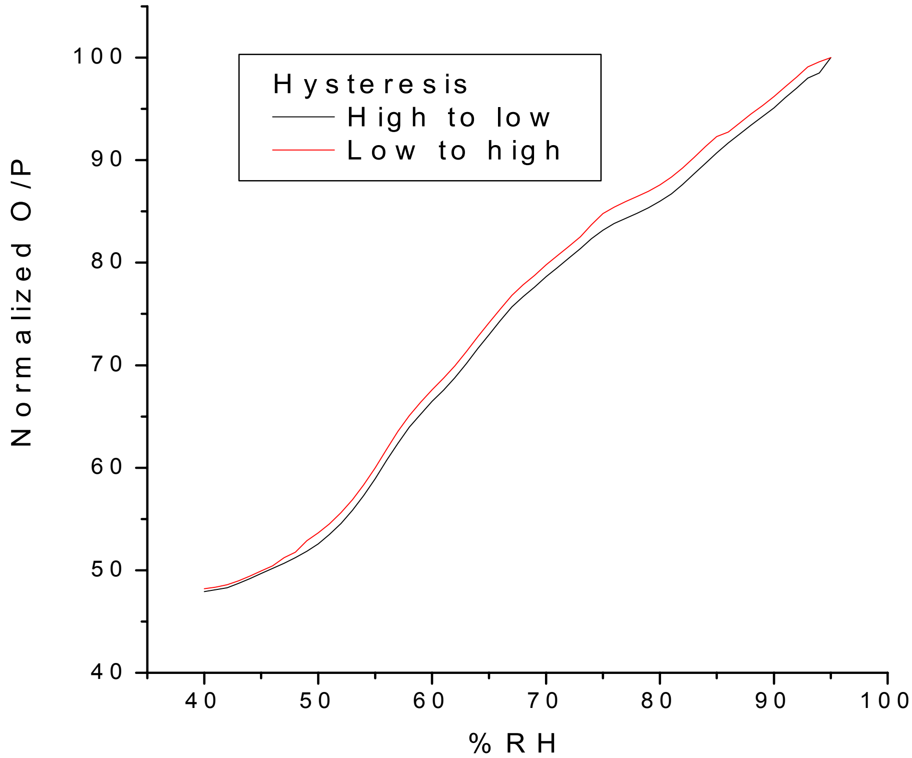

Fig. 8. Variation of humidity for increasing and decreasing cycle of a typical sample for 100% concentration, 3mm length.

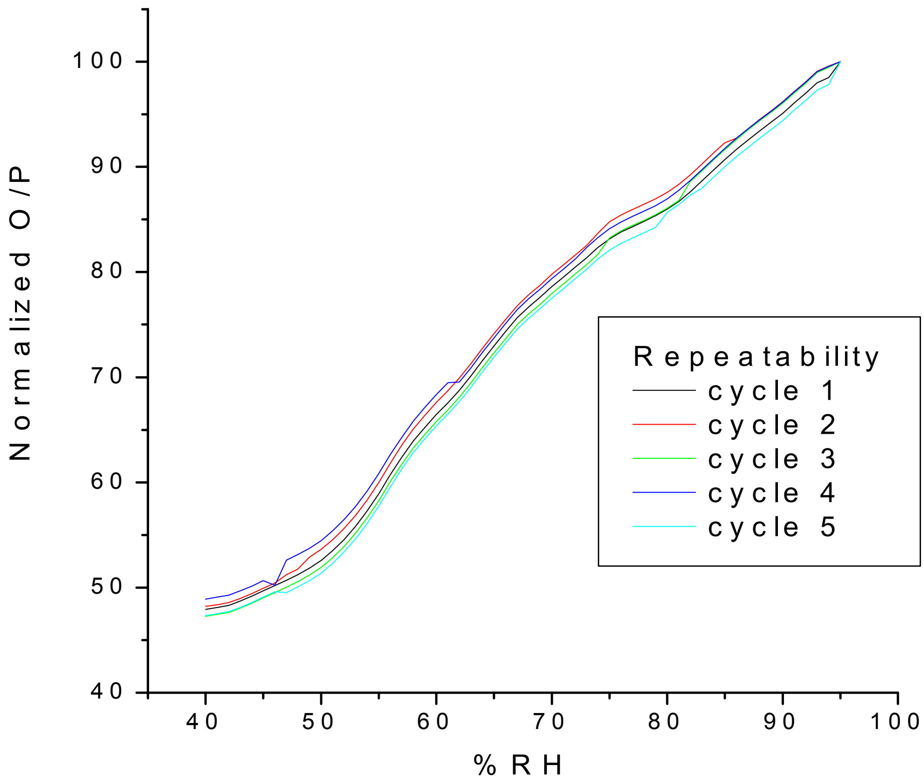

Fig. 9. Variation from cycle to cycle of a typical sample for 100% concentration, 3 mm length.

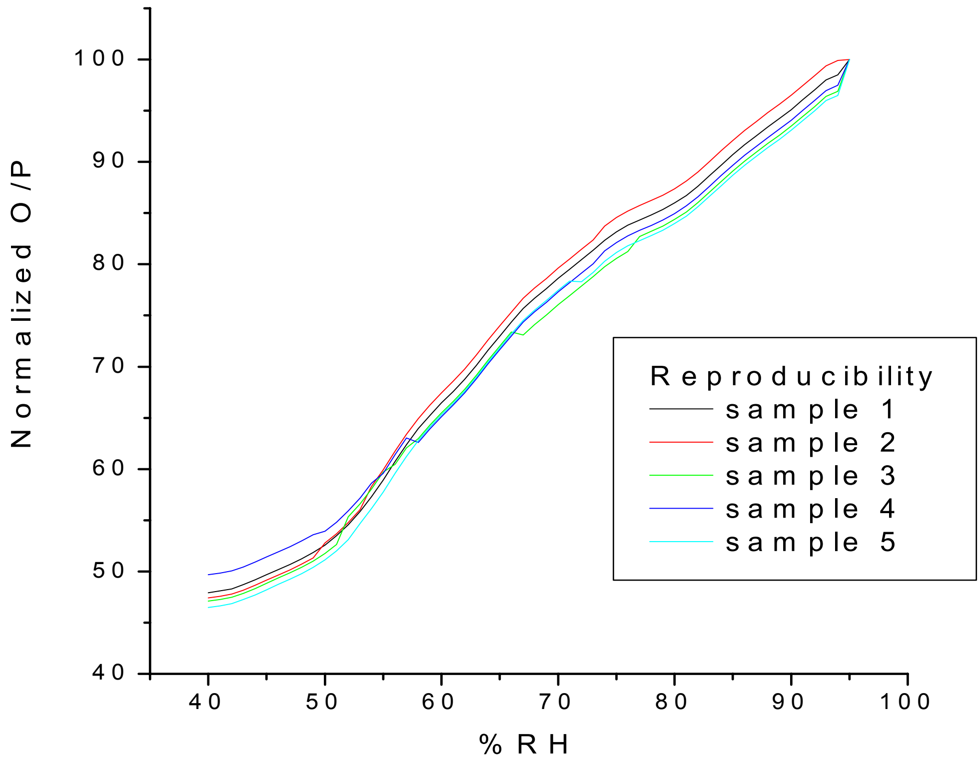

Fig. 10. Variation from sample to sample of a typical sample for 100% concentration, 3mm length.

This sensor is presently expected to be used for air ambient (i.e. normal atmosphere gases and about 30ºC (±5ºC). At room temperature, the operational stability of Biosensor was checked by reusing the sensor for number of cycles. Under identical experimental conditions the sensor shows 7 cycles of reuse with 80% of its initial sensitivity. The relative output intensity was calculated against the number of days to find the shelf life of the biosensor. Under dry conditions at room temperature, the sensor showed high stability (35 days with 80% retention of sensitivity).

4. Conclusions

This is the first time that such biomaterial clad with Ag nanoparticles incorporated in it is used for enhancing humidity sensing. A biosensor for humidity has been fabricated for the range of 40-95% RH. The sensor exhibited a short response of 4-5 and recovery time of 45 sec with high repeatability, reproducibility and a smooth dependence on humidity with little hysteresis effect. The sensor is of low cost and can be easily fabricated.

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Contributions are invited on all aspects of research, development and application of the science and technology of sensors, transducers and sensor instrumentations. Topics include, but are not restricted to:

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- Sensor instrumentation;
- Virtual instruments;
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