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A Study of Relative Humidity Fiber-Optic Sensors

M. Batumalay, S.W.Harun, N. Irawati, H.Ahmad and H. Arof

Abstract — **A humidity sensor made of tapered POF coated with agarose gel or HEC/PVDF detects humidity from the change in the refractive index (RI) of its coating. The RI of the deposited agarose gel or HEC/PVDF coating changes when it swells after absorbing water molecules from the surrounding. Similarly, when a tapered POF seeded with ZnO nanostructure is exposed to ambient humidity, a rapid surface adsorption of water molecules into the ZnO surface occurs. Therefore, the effective RI of its coating, which consists of the thin ZnO nanostrtucture and air, changes with humidity variation. For all of these sensors, the change in the RI of the coating affects the ability of the fiber to modulate light thereby altering the output light intensity. In this paper, the performances of the three coating materials used with tapered fibers to construct humidity sensors are investigated. The results of the experiments show that agarose gel, HEC/PVDF and ZnO based optical fiber sensors are both sensitive and efficient for humidity sensing.**

***Index Terms* -** fiber optic sensor, tapered plastic optical fiber, humidity sensor, relative humidity (RH), agarose gel, hydroxyethylcellulose/ polyvinylidenefluoride (HEC/PVDF) and zinc oxide (ZnO)**.**

I. INTRODUCTION

Plastic optical fibers (POFs) have received wide attention in constructing various optical sensor devices due to many advantages. To widen the range of applications, the development of functional POF in humidity sensing is investigated. In a similar way to temperature, strain or pressure for example, humidity (or moisture content) constitutes one of the most commonly required physical quantities [Yeo, Sun, & Grattan, 2008; Gopel et al., 2008]. The term humidity refers to the presence of water in gaseous form but it is often used to refer to expressions which are related to water vapour characteristics and in the field of measurement, there are various terms associated with such water vapour measurements [Yeo, Sun, & Grattan, 2008].

Fiber sensor researches are focusing in many new areas such as humidity, gases and vapours sensing, medical and chemical analysis, molecular biotechnology, marine and environmental analysis industrial production monitoring, bioprocess control, and the automotive industry. The discovery of refractive index change as an approach used in the Evanescent Wave sensing method (Muto et al., 2003) has generated great interest among researchers to develop high performance devices such as humidity sensors. Humidity sensors are based on reversible absorption of water (H_2O) from the ambient atmosphere into a porous thin-film interferometer that sits on the tapered fiber and changes the refractive index of the thin films and subsequently transforms the lossy fiber into a light guide. Various RH sensors have been explored using a glass silica fiber. For instance Arregui et. al. (2003) uses a tapered silica fiber coated with hydrogels while Bariain etl al. (2000) uses the tapred fiber coated with agarose gel for humidity sensing based on refractive index change. Using on the same approach, humidity sensors was also proposed based on a tapered silica fibers coated with nanostructured films using the ionic self-assembled monolayer (ISAM) deposition technique (Corres et al., 2006; Corres et al., 2007). Many works have also been reported on using a side-polished silica optical fiber coated with a humidity sensitive layer for humidity sensing. Such a sensor was fabricated by means of polishing the flat surface parallel to the fiber axis in order to remove the cladding. The side polishing was realized by first fixing the optical fiber in a rigid holder, forming a rectangular block with fiber extending out from the two end faces of the block orthogonal to the fiber axis. The advantage of this scheme is that the sensing element can be fabricated using inexpensive components and a variety of coating materials can be deposited onto the flat surface of the fiber block. However, the fabrication procedure is very time consuming, dependent upon the design of the fiber block and has limited exposed interaction length. Later, (Gaston et al., 2003; Gaston, Perez and Sevilla, 2004) proposed a humidity sensor based on a single mode, side-polished fiber with a PVA overlay which was tested with 1310nm and 1550nm laser sources and showed different sensing characteristics.

POF offer some advantages over glass optical fiber such as ease of handling, flexibility, low cost test equipment, visible wavelength operating range and high numerical aperture. Even though POF incur higher loss, such effect can be beneficial in the design of sensors as it makes POF based sensors more sensitive. Besides that, POFs have transmission windows in the visible range of 520 to 780nm. Hence, POFs have been relegated to short-distance applications typically of a few hundred meters or less compared with the hundreds of kilometers for glass. POFs have been found many applications in areas such as industrial controls, automobiles, sensors and short data links. Today, a new enthusiasm permeates the plastics side of optical fibers.

In this study, three types of POF tapered sensors are tested where agarose gel, hydroxyethylcellulose/

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polyvinylidenefluoride (HEC/PVDF) and seeded zinc oxide (ZnO) nanostrutures are deposited on the tapered POF and used to measure relative humidity. Their performances at various humidity levels are compared in terms of sensitivity, linearity and limit of detection.

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II. EXPERIMENT

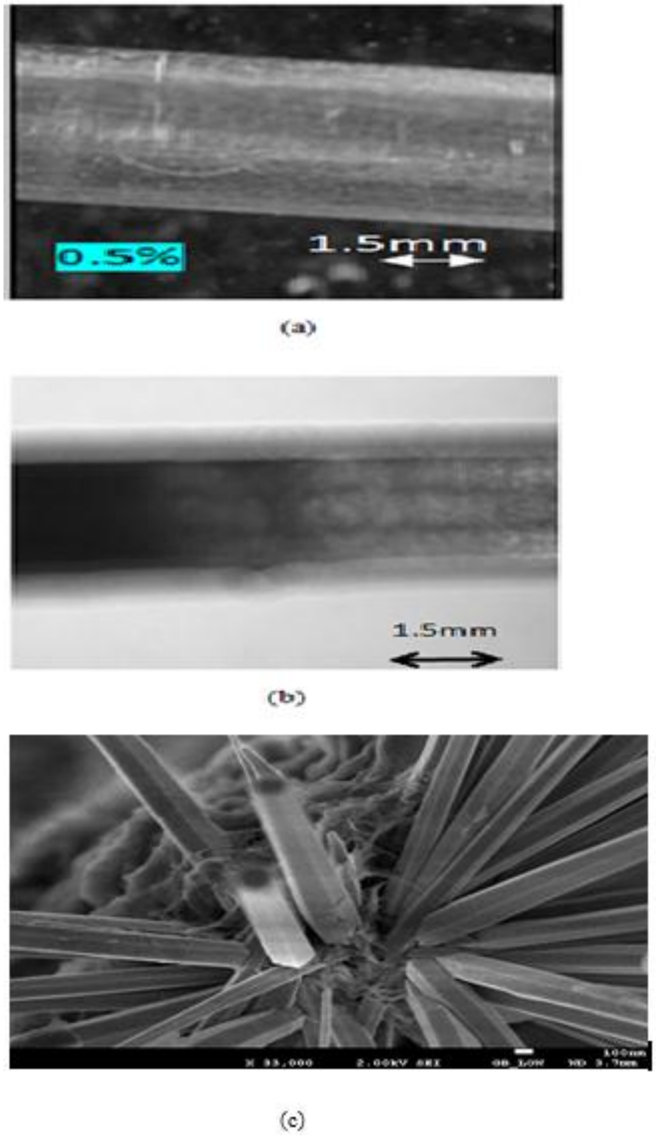
First, a linear type of tapered POF was prepared using acetone, de-ionized water and sand paper in accordance with chemical etching technique. The POF has an overall cladding diameter of 1 mm, a numerical aperture of 0.51 and an acceptance angle of 61°. The refractive index of the core and cladding are 1.492 and 1.402 respectively. The acetone was applied to the POF using a cotton bud and neutralized with de-ionized water. The acetone reacted with the surface of the polymer to form milky white foam on the outer surface of the cladding which was then removed by the sand paper. This process was repeated until the tapered fiber had a stripped region waist diameter of 0.45 mm. Beres et al [2011] observed that tapered fibers with waist diameters in the range of 0.40mm to 0.50mm showed good sensitivity to refractive index variation whereas those with waist diameters above 0.55mm and below 0.30mm did not demonstrate substantial sensitivity. The total length of the tapered fiber for this section was fixed at 10 mm. Finally, the tapered POF was cleansed again using the de-ionized water.

Then agarose gel, HEC/PVDF and ZnO were prepared to coat the tapered POF with. As mentioned by Stellan et al. [1981] the porosity of agarose gels decreases as the concentration of agarose increases. Agarose has a high porosity which allows the gel to absorb moisture and perform as humidity sensor. The agarose gel used is based on swelling nature of hydrophilic materials which causes refractive index changes in accordance with humidity and modulates the light propagating through the fiber. In an earlier work, we reported that fiber with agarose gel of 0.5% weight content shows higher sensitivity in comparison with 1% and 1.5% due to the effect of pore size [Batumalay et al., 2014]. Therefore, agarose powder obtained from Sigma Aldrich (no. A6013) was dissolved in water, in proportions of 0.5% in weight for this experiment. The mixture was heated to 50°C. A small portion of the mixture was deposited on the tapered area of the fiber. Then, the fiber was left to dry for a day. Fig. 1 (a) shows the microscopic images of agarose gel deposited on the tapered POF.

For the HEC/PVDF, 1 g of PVDF powder (Mw = 275,000) was dissolved in 120 ml dimethyl form amide (DMF) at 90^{o}C in water bath. The solution of PVDF was cooled down to room temperature, and then 4g of hydroxyethyl cellulose (HEC) was added to the solution. The mixture was continuously stirred at room temperature for about 10 hours to make it completely homogenous as described by Muto et al [2003]. Both ends of the POF were held and straightened on translation stages before the HEC/PVDF was deposited onto the tapered fiber. Then the HEC/PVDF mixture was slowly dropped onto the tapered region of the fiber using syringe and left to dry for 48 hours. Fig. 1(b) shows the microscopic image of tapered POF coated with HEC/PVDF composite.

As for ZnO nanostructures, two major steps were carried out. First, the seed layer was deposited on the fiber to grow the ZnO nanostructures on using a simple manual dip coating technique. The seeded solution was prepared by dissolving zinc acetate dehydrate ($Zn(CH_3COO)_2{\cdot}2H_2O$) as a precursor in isopropanol with a molarity of 0.025 M. The solution was stirred at 60° C for 2 hours at ambient temperature to yield a clear and homogenous solution. Then, the solution was cooled down to room temperature for the coating process. The fiber was manually dipped into the seeding solution and dried at 50° C to evaporate the solvent and remove the organic residuals. This coating and drying procedure was repeated 5 times to increase the thickness of the fibers. Following this, the growing solution was prepared by dissolving 0.01M zinc nitrate hexahydrate ($Zn(NO3)_2.6H_2O$ and 0.01M hexamethylenetetramine (HMTA) in 100ml deionized water. The deposition process of ZnO nanorods on the fibers was performed using sol-gel immersion method by suspending the seeded-ZnO fibers in the growing solution at 60°C for 15hours. Fig. 1 (c) shows the FESEM images with ZnO nanostructures grown on seeded tapered fiber.

Fig. 2 shows the experimental setup of the proposed sensor used to detect relative humidity using the fabricated tapered POF with and without HEC/PVDF composite. The setup consists of a light source, an external mechanical chopper, the proposed sensor, 1365 data logging humidity-temperature meter, a highly sensitive photo-detector, a lock-in amplifier and a computer. The input and output ports of the tapered POF are connected to the laser source and photo-detector, respectively. He-Ne laser operating at a wavelength of 633 nm with an average output power of 5.5 mW is used in the experiment because POFs have transmission windows in the visible range of 520 to 780nm. The light source is chopped at a frequency of 113 Hz by a mechanical chopper to prevent the harmonics from the line frequency, which is about 50 to 60 Hz from influencing the results. The He-Ne light source is launched into the tapered POF placed in a sealed chamber with a dish filled with saturated salt solution. The output light is sent to the silicon photo-detector (818 SL, Newport) to be converted into electrical signal. Then the electrical signal together with the reference signal from the mechanical chopper is fed into the lock-in amplifier (SR-510, Stanford Research System). Finally, the output of the lock-in amplifier is sent to a computer through an RS232 port interface. The signal is processed using Delphi software. The reference signal from the chopper is matched with the input electrical signal from the photo-diode to remove the noise generated by the laser source, photo-detector and the electrical amplifier in the photo-detector. In this experiment, the performance of the proposed sensor is investigated for various relative humidity ranging from 50 to 80% using 1365 data logging humidity-temperature meter.



(a)

(b)

(c)

Fig 1. Microscopic images of (a) tapered POF coated with agarose gel of 0.5% weight content (scale: 1cm: 1.5mm), Fig. 1 (b), tapered POF coated with HEC/PVDF composite (scale: 1cm: 1.5mm) and Fig. 1 (c), FESEM image of coated with ZnO nanostructures grown on seeded tapered POF.

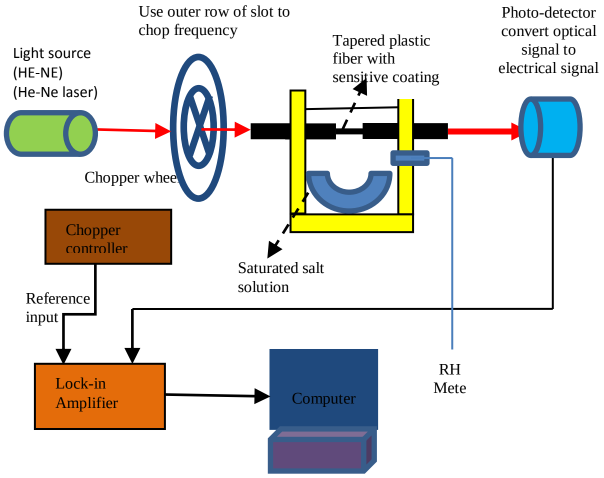


Fig 2. Experimental setup for the proposed relative humidity sensor using a tapered POF with sensitive coating materials.

III. RESULTS AND DISCUSSIONS

The tapered bare fiber without coating was first tested to measure the relative humidity where the sensitivity of the sensor is obtained at 0.0028 mV/% with a slope linearity of more than 93.29% and limit of detection of 45.45% as shown in Fig. 3. The limit of detection was calculated by dividing the standard deviation with the sensitivity, and thus the system is more efficient when the limit of detection is lower. The sensitivity of the bare fiber is the lowest when compared to that of the tapered fibers coated with agarose, HEC/PVDF and ZnO Nanostructure.

The same figure also shows the variation of the transmitted light against the relative humidity for tapered fiber coated with agarose gel. It is observed that the intensity of the transmitted light through the agarose coated tapered fiber decreases as relative humidity increases from 50 to 80%. Its sensitivity is 0.0228 mV/% with a slope linearity of more than 98.36% and a limit of detection of 0.921%. In short, the tapered fiber coated with hydrophilic material such as agarose gel demonstrates a higher sensitivity to humidity change. Agarose has a high porosity which allows the gel to absorb moisture that consequently changes its RI and the modulation of light propagating through the fiber. It is observed that the output voltage is found to be decreasing, as the RH increases. According to Lee et al. [2007] the RI value of agarose gel varies from 1.52 to 1.54 when RH changes from 20% to 80%. The humidity sensitive layer of the composite has an RI value which is higher than that of the core creates a lossy waveguide which leads to decrease in output voltage.

The tapered POF coated with HEC/PVDF also displays a linear relationship between relative humidity and output intensity. The output voltage from the photo-detector shows that the transmitted light intensity linearly increases as the relative humidity rises from 50 to 80%. The probe produces a sensitivity of 0.0231 mV/% with a slope linearity of more than 99.65% and a limit of detection of 5.75%. According to Muto et al. [2003], the refractive index of hydroxyethylcellulose (HEC) film, which was measured using Abbe's refractometer, changed from 1.51 in the dry state to 1.48 in humid air with 80% RH. The humidity sensitive layer of the composite has an RI value which is higher than that of the core in dry state. This situation creates a lossy waveguide and as the cladding layer hydrates, the RI value falls below that of the core and increases the intensity of light propagating through the core. The lower limit of detection for the probe with HEC/PVDF composite in comparison with the bare fiber also shows that the system is more efficient.

Finally, the performance of the tapered POF coated with ZnO nanostructures is also studied. As shown in fig. 3, the intensity of the transmitted light decreases linearly with the increase in relative humidity. It is found that the sensitivity of tapered POF grown with seeded ZnO is 0.0258mV/% with a slope linearity of more than 95.48% and limit of detection of 0.143%. Evidently seeding the tapered fiber with ZnO nanoparticles as seeds has enhanced the growth of nanorods. These rods absorb more water and increase the sensitivity of the sensor. In addition, the seeding technique reduces the limit of detection, which indicates that the sensor system is more efficient. According to Liu et al., the effective refractive index (RI) of ZnO composite varies from 1.698 to 1.718 as the relative humidity changes from 10-95% [2012]. As the ZnO coating is exposed to humidity, rapid surface adsorption of water molecules occurs. The increase in water molecules being absorbed by ZnO layer increases the RI of the effective coating of the fiber which leads to a larger leakage of light [Liu et al., 2012]. As a conclusion, it is found that the sensitivity of the sensor is significantly improved by the seeding technique.

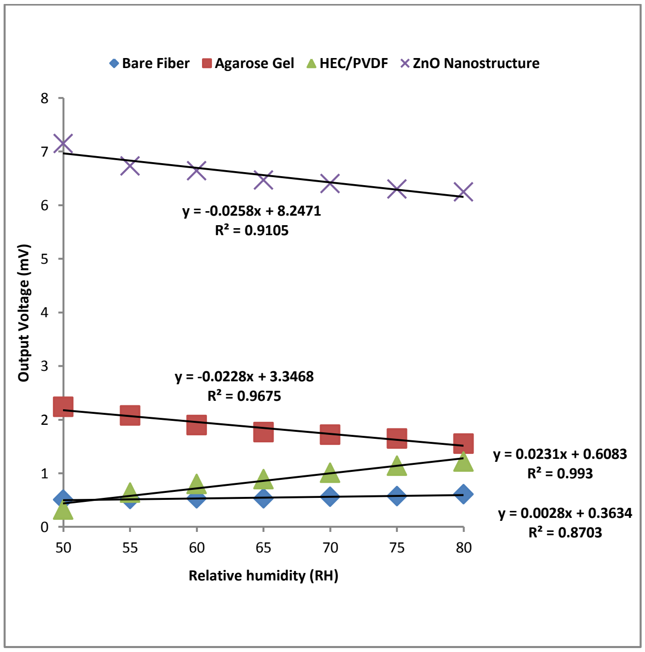


Fig. 3: Output voltage against RH for the bare fiber, Agarose, HEC/PVDF and ZnO Nanostructure

Reversibility of the results is another important factor to consider when measuring input-output relationship of any sensor system. Thus, the next step is to validate the parameters obtained as the RH is varied from 80% down to 50%. The measured output intensity as a function of RH is recorded for two different runs and compared. As noted in Fig. 4, the maximum difference between the two runs for agarose gel is about ±0.05mV, which is acceptable for a full-scale output of 2.25mV. For the HEC/PVDF composite, the maximum difference between the two runs is about ±0.05mV, which is acceptable for a full-scale output of 1.25mV and for the ZnO nanostruture, the maximum difference between the two runs is about ±0.05mV, which is also acceptable for a full-scale output of 7.15mV.

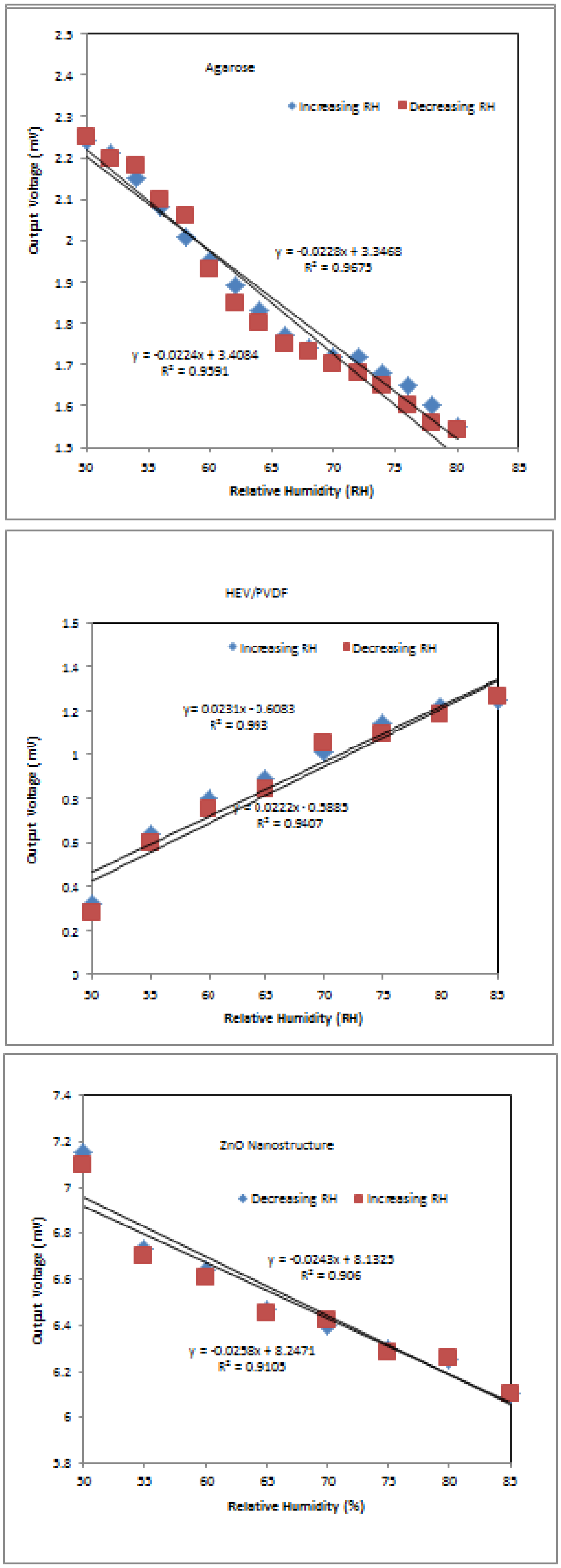


Fig. 4: The reversibility of the results obtained for two different runs (Relative Humidity).

The performance characteristic of the proposed sensor is summarized in Table 1. In measuring RH within a range of 50 to 80%, the sensor coated with agarose gel displays a sensitivity of 0.0228 mV/% with a slope linearity of more than 98.36%. Its limit of detection is 0.927%.

We believe other hydrophilic materials can also be used to coat the tapered POF as long as its refractive index can change in response to humidity variation. The tapered POF coated with HEC/PVDF composite shows a sensitivity of 0.0231 mV/% and a linearity of more than 99.65%. Its limit of detection is calculated at 5.75%. Since the composite has an RI value which is higher than that of the fiber core in dry state, it behaves as a lossy waveguide. As the cladding layer hydrates in the presence of moisture, its RI value falls below that of the core and increases the intensity of light propagating through the core. In short, the rise in humidity level reduces the effective refractive index of the composite cladding thus allowing more light to be transmitted. However, the best sensitivity belongs to the tapered POF coated with seeded ZnO nanostructure. This sensor provides the highest sensitivity of 0.0258mV/% with a slope linearity of more than 95.48% and limit of detection of 0.143%. This sensor is observed to be sufficiently stable for RH changes with standard deviations of 0.0037mV as the measurement is taken within 100s.

Table 1 Performance of the proposed sensors.

	Bare fiber	Fiber with agarose 0.5% weight content	Fiber with HEC/PVDF composite	Fiber with ZnO nanostructure
Sensitivity (mV/%)	0.0028	0.0228	0.0231	0.0258
Linearity (%)	93.29	98.36	99.65	95.48
Std Deviation (mV)	0.1509	0.021	0.133	0.0037
Limit of detection (%)	45.45	0.921	5.75	0.143

IV. CONCLUSION

Simple fiber optic RH sensors have been succesfully demonstrated to measure RH change in the range of 50 to 85%. The tapered fiber sensor with agarose gel coating of 0.5% weight content shows a sensitivity of 0.0228 mV/%. Its slope linearity is more than 98.36% and its limit of detection is 0.927%. The output intensity of the one coated with HEC/PVDF composite increases linearly with RH. Its sensitivity, linearity and limit of detection are 0.0231 mV/% , 99.65% and 5.75% respectively. The tapered POF seeded with ZnO nanostructure provides the highest sensitivity at 0.0258mV/% with a slope linearity of more than 95.48% and limit of detection of 0.143%. Removing the cladding of the tapered POF allows the sensitive material to function as a passive cladding. The sensitive materials play an important role in the RH sensing. Consequently, its refractive index can

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influence the amount of power loss as the signal propagates through the tapered region. This is attributed to the difference in refractive index between the core and cladding that influences the amount of light confined inside the core. The results show that the sensitive coating materials have successfully enhanced the performance of these POF sensors and applicable for RH detection.

In conclusion, the best sensitivity belongs to the tapered POF coated with seeded ZnO nanostructure where the system provides the highest sensitivity of 0.0258mV/%. The lowest limit of detection for the tapered POF coated with seeded ZnO nanostructure in comparison with the bare fiber, tapered fiber with Agarose 0.5% and HEC/PVDF composite shows that this system is more efficient. This sensor is also observed to be sufficiently stable for RH changes with the lowest standard deviations of 0.0037mV as the measurement is taken within 100s.

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