

Optical Humidity Sensing Characteristics of Ag-Polyaniline Nanocomposite

Madhavi V. Fuke, Anu Vijayan, Prajakta Kanitkar, and R. C. Aiyyer

Abstract—Optical transmission type humidity sensor is fabricated using spin coated films of Ag-Polyaniline nanocomposite. The material is coated layer-by-layer to get films of various thicknesses (20–40 μm). Relative humidity (RH) was generated by the standard two temperature method from 15 to 100% RH. The sensor shows quick response of 7 s and a fast recovery time of 50 s with better sensitivity for higher thickness. The Ag-Polyaniline nanocomposite is characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), UV-Visible spectroscopy (UV), and Fourier Transform infrared spectroscopy (FTIR). The repeatability and cycle to cycle variation is studied and found to be encouraging.

Index Terms—Ag-Polyaniline nanocomposite, optical sensor, relative humidity.

I. INTRODUCTION

THE humidity control and monitoring is of increasing importance in environment, in agro base applications like harvesting, watering the plants, grain storage, preparations of jams and gellies, paper manufacturing, medical applications, and some industrial chemical industrial sectors. Among all the available methods, the one based on optical transmission is simpler and easy to carry out, comparatively new and needs low-cost equipment for studying humidity sensors. Measuring and evaluating changes in the refractive index of the sensing material induced by water vapor is also carried out. High accuracy and more convenience can be achieved from measurement of the optical phase change by optical interferometry [1]. Over many years, extensive study of the relative humidity measurement has been done and there is a remarkable progress in the development of relative humidity sensors [2]–[6]. Commercial humidity sensors are mostly comprised of either polymer films [7], [8] or porous ceramics [9], [10]. These sensors are based on the principle of change in electrical resistance as a function of relative humidity. The optical sensors are attracting increasing interest due to their inherent characteristics such as immunity to electrical noise, ease of fabrication, and the possibility of real-time monitoring and remote sensing. Polymer

claddings and inorganic oxides as sensing layers have been applied and presented lately in fiber-optic [11], waveguide [1], and Fabry Perot [12] interferometric type devices. Nowadays, nanocomposites are being used as humidity sensing materials. The nanoparticles incorporated within a matrix act as suitable candidates for sensing. The nanoparticles have a large surface to volume ratio making these materials potentially attractive for sensing purposes because, in principle, they should provide innumerable surface sites for physisorption of water molecules. The possibility of using the nanocomposites as humidity sensors are explored by Pal *et al.* [13] and Das *et al.* [14]. Nano-like magnesium oxide films and its significance in optical humidity sensing is reported by Shukla *et al.* [15].

Polymers are identified as potential candidates for practical applications because they are compatible with oxides and ceramics, because of their low-cost, flexibility, light weight, and easy processibility and they can be used at room temperatures. A variety of polymers are being used in optical sensors such as silicones, poly (vinyl chloride), poly (tetrafluoro ethylene), nafion, nylon, agarose, sol gels, etc. [16]. In general, polymers provide useful mechanical properties to the sensor design. Among the conducting polymers, Polyaniline has received great attention owing to its simple method of synthesis, stability against environmental conditions, ease of doping with protonic acids, and practically usable electrical conductivity [17], [18]. Parvatikar has reported humidity sensing by using PANI/Co₃O₄ composites and PANI/CeO₂ composites [16], [19]. An optical fiber-based humidity sensor using Cobalt-Polyaniline nanocomposites has been reported by Vijayan *et al.* [20].

Here, a simple, low-cost, optical transmission-based humidity sensor is reported using Ag-Polyaniline nanocomposite dispersed in Acetonitrile to form films of various thicknesses. The material characterization is done using scanning electron microscopy (SEM), X-ray diffraction (XRD), UV-Visible spectroscopy (UV), and Fourier transform infrared spectroscopy (FTIR). The synthesized nanocomposite is successfully utilized as a humidity sensor for a broad range of humidity, ranging from 15% to 100% RH. The main aim of this paper is to realize a humidity sensor with a large operational range, good sensitivity, repeatability, reproducibility with low hysteresis

II. EXPERIMENTATION

A. Synthesis of Ag-Polyaniline Nanocomposite

For synthesizing Ag-Polyaniline nanocomposite, AR grade chemicals, and monomer were used. For making solutions, double distilled water was used. The process of polymerization was initiated by addition of the oxidizing agent drop-by-drop,

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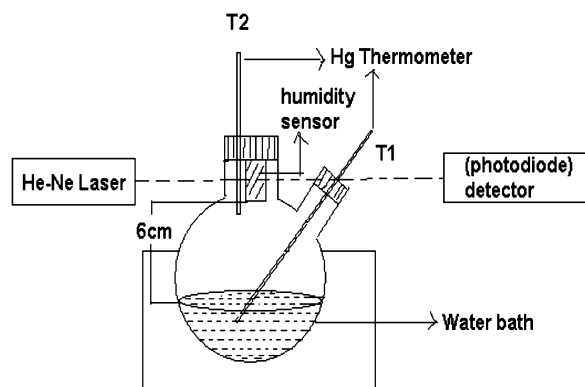


Fig. 1. Schematic of two temperature method for the measurement of relative humidity.

Ag nano particles were synthesized by a well known electrochemical method, $(\text{NH}_4)_2\text{S}_2\text{O}_8$ (dissolved in minimum amount of water) in an acidified solution of monomer was added with synthesized silver nanoparticles [21] under constant stirring between 0 and 5 °C. During *in situ* polymerization, Para toluene sulphonic acid (p-TSA) was used as a dopant. The monomer to oxidizing agent ratio was kept as 1:1. After complete addition of the oxidizing agent, the reaction mixture was capped by stopper and kept under stirring for 24 h. The greenish black precipitate of the polymer was isolated by filtration. It was conditioned by washing and then dried for 12 h in an oven kept at 60 °C. Thus, the pure Ag-Polyaniline nanocomposite in powder form is formed. This powder is dispersed in acetonitrile to get Ag-Polyaniline nanocomposite solution and this solution was further used for experimentation.

B. The Sensor Fabrication and its Characterization as a Humidity Sensor

For fabricating a sensor, the solution was spin coated layer-by-layer on a glass substrate of 5 mm × 5 mm fixed area with an optimized speed to get uniform films. The films of various thicknesses (20–40 μm) are casted by spinning 0.1 cc of Ag-Polyaniline solution each time and thicknesses of the films are measured by using a Light Section Microscope (BK 70 × 50, VEB Carl Zeiss Jena, Germany).

The above prepared films were examined for their sensitivity towards humidity measured by the standard two temperature method.

A closed humidity system similar to that of cutting [3] is used for the two temperature method (Fig. 1). It consists of a closed flask, with two necks for inserting thermometers (range-10 °C to 60 °C with an accuracy of 0.5 °C), half filled with water (total volume 500 ml). The flask is kept in a thermocol container. External container is filled with ice to the equal level of water in the flask. The temperature of the system is adjusted by mixing ice and water as required. Thus, the water inside the flask can be kept at the required temperature (T_1). The sensor is mounted inside the flask at a height of 6 cm from the level of water and the temperature of the sample (T_2) is measured with a thermometer, which is placed at the height of the sample under study. It is to be noted that the temperature of the sample changes by 3 °C to 6 °C during the experiment.

For calculating the relative humidity inside the chamber the equation given below is used which gives the relative humidity (RH) of the air in the system

$$\%RH = E_w(T_1)/E_w(T_2) \times 100$$

where

$E_w(T_1)$ the saturated vapor pressure at the temperature of the water bath (T_1);

$E_w(T_2)$ the saturated vapor pressure at the temperature of the sample element (T_2).

From the CRC manual of chemistry [22], the values of the saturated vapor pressure are obtained. By adjusting the temperature of water inside the flask, with ice and water mixture from room temperature to 0 °C, ranges of RH values are obtained.

The plane of the film is placed perpendicular to a He-Ne (632.8 nm) laser beam. The output transmitted through the sample (films) is measured using simple photovoltaic detector and is normalized with respect to the lowest humidity. Normalized output is plotted with respect to the relative humidity. The sensitivity is defined as the change in transmitted output (mV) per unit change in RH% i.e. $\delta(\text{mV})/\delta(\text{RH}\%)$ [6].

The response time is measured as the time required for the stable output when the sample is subjected to change in RH from lower value (Room RH) to a higher value (100%RH) as a step function. The response time is also observed for the direct transition from 15%RH to air ambient (55%RH). The recovery time is measured after exposing the sensor directly to the air ambient from 100%RH by removing the sample from the test chamber, till the sensor regains the original output value. The recovery time is also observed for direct transition from 100%RH to 15%RH.

For measuring hysteresis of the sensor, first the humidity of the chamber is reduced to 15%RH and then, increased to maximum humidity, i.e., 100%RH. The maximum difference in the output of the two cycles at any particular RH is measured as the hysteresis. Four cycles are performed on a sample to study the reliability of the sensor.

C. Material Characterization

X-Ray diffraction was performed with a Philips X-Ray diffractometer ($\text{CuK}\alpha$ as radiating wavelength). FTIR spectra of the samples were recorded on a Perkin Elmer-Spectrum 2000 spectrophotometer operated between 400 and 4000 cm^{-1} in a KBr medium. An Analytical Scanning Electron Microscope JOEL JSM 6360A was used to investigate the powder morphology of the composites in the form of film. A UV-Vis spectrum of the nanocomposite in acetonitrile was recorded using Perkin Elmer spectrophotometer in the range of 200–800 nm.

III. RESULTS AND DISCUSSION

A. Material Characterization

i) *SEM of Ag-Polyaniline Composite*: The SEM micrographs of Ag-Polyaniline the films of various thicknesses are as shown in Fig. 2. With increasing thickness, the pore size increases. At

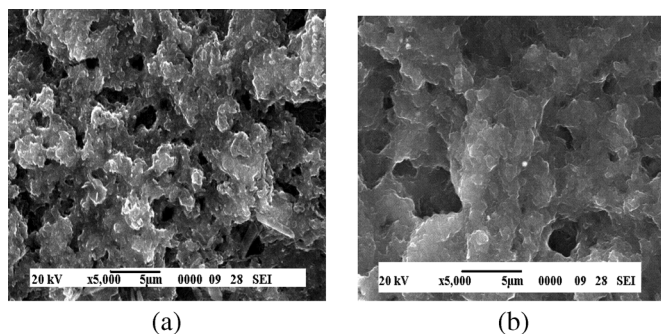


Fig. 2. SEM of Ag-Polyaniline nanocomposite for the thicknesses of (a) 20 μm and (b) 40 μm .

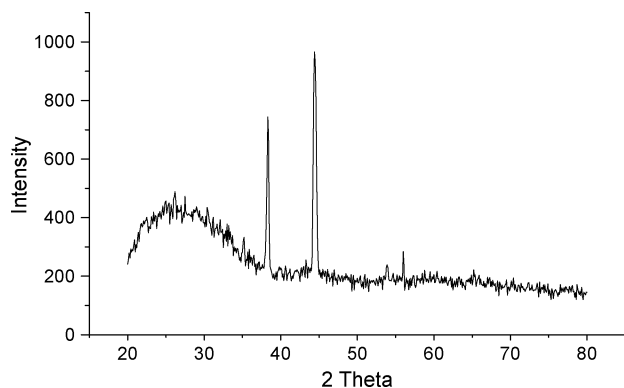


Fig. 3. XRD spectrum of Ag-Polyaniline nanocomposite.

the thickness of 40 μm , agglomeration of the nanocomposite is seen with practically the least number of pores. For lower thickness, porosity exists whereas for higher thickness the composite shows agglomeration of polymer. Such composites are likely to facilitate adsorption of water vapors because of the pores.

ii) *XRD of Ag-Polyaniline Composite*: XRD of Ag-Polyaniline nanocomposite (Fig. 3) show peaks having 2θ values of 26° , 56° which corresponds to the peak for Polyaniline. Especially the peak at 26° reveals the crystalline nature of the composite resulted due to the sequential growth of Polyaniline chain on the silver nanoparticles [23]. The composite is having peaks for both: Polyaniline and Silver. The peaks for pure Ag and Ag composite are available. The peak of silver sulphide is seen at 2θ values 35.13° with corresponding plane (022). 53.9° , 2θ value gives the peak of AgCl in (201) plane. Peaks corresponding to 2θ values 38.2° and 44.2° reveal the crystalline nature of silver in planes (111) and (200), respectively.

iii) *UV-Vis of Ag-Polyaniline Composite*: The UV-Vis spectrum of Ag-Polyaniline nanocomposite is shown in Fig. 4. Silver nanoparticles show an intense absorption peak around 400 nm originating from the surface plasmon absorption of nanosized silver particles. The comparative study of absorption spectra of pure Polyaniline and Ag-Polyaniline shows that the shoulder peak at 320 nm is suppressed and the peak of 420 nm is enhanced due the presence of Ag nanoparticles in the nanocomposite.

iv) *FTIR of Ag-Polyaniline Composite*: Fig. 5 elaborates the FTIR spectrum of Ag-Polyaniline nanocomposite. The peaks at 1126.58 cm^{-1} and 684.46 cm^{-1} give C-H in plane and C-H

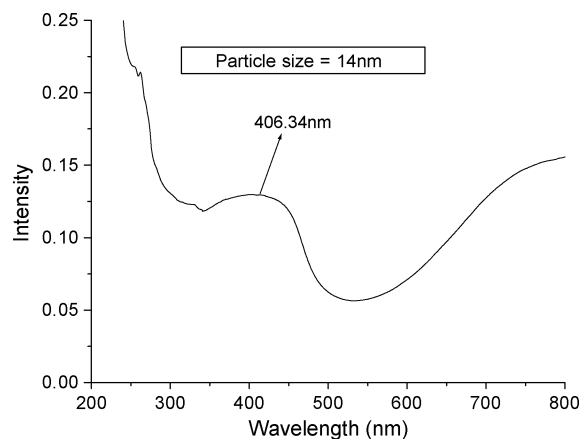


Fig. 4. UV-Visible spectrum of Ag-Polyaniline nanocomposite.

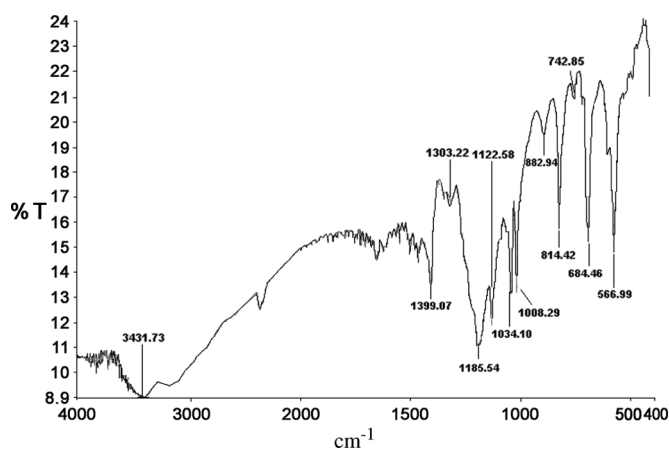


Fig. 5. FTIR spectrum of Ag-Polyaniline nanocomposite.

out of plane bending vibrations, respectively. The characteristics of paradiistributed aromatic rings indicating polymer formation shows the peak at 814.42 cm^{-1} . Aromatic C-N stretching indicating appearance of the secondary aromatic amine group is observed at 1303.22 cm^{-1} . Benzoid ring and quinoid ring modes are of particular interest and the bands in the vicinity of 1399.07 and 1588.16 cm^{-1} corresponding to the Benzoid ring and quinoid ring modes, respectively. The presence of these bands clearly shows that the polymer is composed of insulating and conducting phase [24], [25]. The metal oxygen stretching frequency of Ag-O is at 567 cm^{-1} . Band at 2928.28 cm^{-1} is assigned to aromatic C-H stretching vibration. The presence of all these characteristics bands confirms that the polymer is in the conducting ES phase.

B. Humidity Response for Ag-Polyaniline Nanocomposite

Typical response of the sensors as a function of relative humidity, i.e., the change in transmitted light intensity with RH% for various thicknesses is exhibited in Fig. 6. The sensor is characterized by varying the relative humidity at a room temperature of 25°C – 27°C . In general, it is observed that the transmission decreases with increase in RH in the range 20% to 100%RH.

All the sensors exhibit roughly three regions of sensitivity and have very low sensitivity at lower humidity (Region I roughly

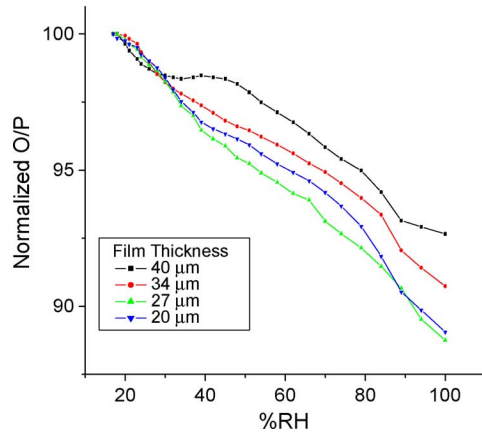


Fig. 6. Sensitivity curve for the normalized output with respect to the relative humidity for different thicknesses.

TABLE I
REGION WISE SENSITIVITY FOR DIFFERENT THICKNESSES OF
Ag-POLYANILINE NANOCOMPOSITE

Thickness of Ag – Polyaniline film(μm)	RI of Ag-Polyaniline	Region I (mV/RH)	Region II (mV/RH)	Region III (mV/RH)
40	1.49	79.4(17-34%RH)	80.4(35-77%RH)	193.6(78-100%RH)
34	1.45	81.9(17-34%RH)	122.5(35-70%RH)	239.6(71-100%RH)
27	1.40	93.5(17-34%RH)	300(35-73%RH)	377(74-100%RH)
20	1.35	86.6(17-34%RH)	168.3(35-74%RH)	285.1(75-100%RH)

17% to 34%RH- on an average 85 mV/RH), in the middle range (35% to 74%RH) the sensitivity increases and at still higher humidity (75 °C to 100%RH) the sensitivity increases further, as shown in Table I.

The maximum sensitivity is observed for films having thickness of 27 μm. The mechanism of humidity sensing is explained. The water vapor molecules are chemisorbed through a dissociative mechanism by which two surface hydroxyl groups per water molecule are formed. The layer of hydroxyl groups is formed on the surface of the film at lower humidity [19]. This does not change the transmission of light intensity through the film within the limits of measurements (0.1 mV). Therefore, at lower relative humidity, the sensor response is poor. At a higher humidity (Region II), the water molecules get adsorbed on the wall of the pores, as well as second or multilayers of water molecules are formed on the surface of the film. The light through the film gets absorbed or scattered in proportion to the deposition of water molecules on surface of the film or the pore walls, decreasing the output voltage. In Region III, the output decreases because all the pores and film surfaces are full with water molecules. The change in the output is attributed to the absorption/scattering of light because of the water meniscus formed on the film.

The decrease in the output voltage with increase in the relative humidity can also be attributed to a mobility of Ag ions which are loosely attached to the polymer by weak Van der

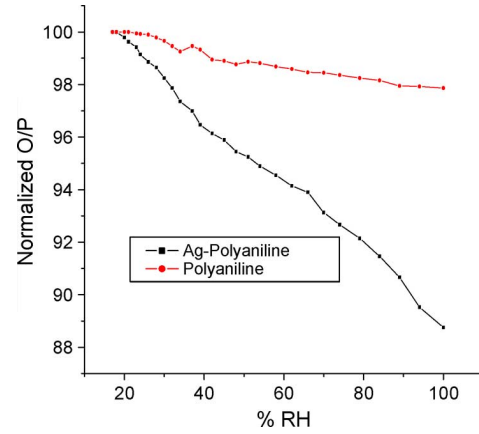


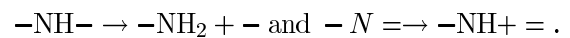
Fig. 7. Study to compare humidity response of Polyaniline and Ag-Polyaniline.

Waals' forces of attraction. At low humidity, the mobility of Ag is restricted because under dry conditions the polymer chains would tend to curl up into compact coil form. On the other hand, when water molecules get absorbed compact foils uncurl and are transferred to a straight chain changing the transmission properties of the film.

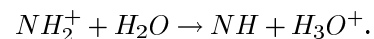
With increase in thickness, the surface morphology of the films is changing. From SEM, it is observed that the porosity is higher at lower thicknesses and hence gives higher sensitivity. The changes in surface morphology are creating more pores/channels for absorption of water. At higher thickness, more water is absorbed which also absorbs the incident light. This situation favors the output voltage to decrease [16].

Fig. 7 shows the comparative study of Polyaniline and Ag-Polyaniline nanocomposite for humidity sensing. Ag-Polyaniline is more sensitive to humidity in comparison to Polyaniline alone. This clearly shows that Ag nanoparticles play an important role in enhancing the humidity sensing.

The mechanism of oxidation and reduction decides the increase or decrease in the output [26], [27]. The Polyaniline structure contains of two basic forms, i.e., the nonoxidized (reduced) and oxidized structures, due to polymerization by strong oxidant ((NH₄)₂S₂O₈). The Polyaniline synthesized by this way can be regarded as p- type doping. Both the forms can be protonated due to the unbonded electron pair on the nitrogen atom



The hopping of electrons from protonated reduced form (–NH₂ + –) to the protonated oxidized form (–NH⁺ =) is the dominant process of sensing. The following acid-base reaction describes the transfer of proton from polymer in presence of water molecule which is studied by NMR



The variation of output of Ag-Polyaniline nanocomposite is the result of the redox reactions in the presence of the humidity.

The fast response of the sensors may be due to the fast penetration of water molecules into the film. The slow desorption

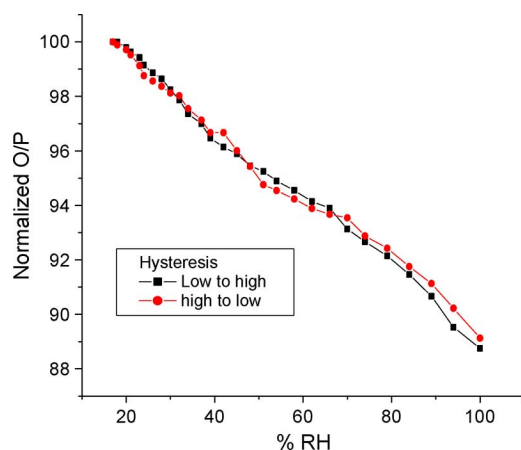


Fig. 8. Response curve with increasing and decreasing humidity for the Ag-Polyaniline nanocomposite for 27 μm thickness.

process and capillary forces results in the slow recovery in the response [6].

Fig. 8 shows the sensor hysteresis between absorption and desorption process. The results indicate that the desorption rate of water molecules in the Ag-Polyaniline film is slower than the adsorption one. Hysteresis is observed to be nearly 1% for all of the thicknesses which is defined as the maximum difference in the two outputs (increasing and decreasing cycle) at the same RH level. Slow desorption of the water from the pores of the capillary results in the hysteresis, in the response at lower RH. The capillary condensation occurs at higher humidity and forms a meniscus over the capillaries of the film. The hysteresis and nonlinearity in the response can be attributed to this. Here, 1%–2% hysteresis is shown for 27 μm thickness, as in Fig. 8.

Two different humidity chambers of 95%RH and 15%RH are prepared which is used to measure the response time of the fabricated Ag-Polyaniline humidity sensors. A sensor which is in one column (high or low humidity) is moved into the other column as soon as possible in the same constant temperature chamber and during the adaptation of sensors change in output voltage is recorded as a function of the time.

The repeatability and reproducibility of the sensor turned out to be excellent and that was checked for four cycles and samples, respectively. The repeatability and reproducibility was found to be good for all the thicknesses and a result for 27 μm thickness is shown in Figs. 9 and 10, respectively. About 2%–3% uncertainty from cycle to cycle and uncertainty of about 3%–4% from sample-to-sample is observed

The sensing response time of layer-by-layer (LbL) nano-assembly for deposition of ultrathin poly (anilinesulfonic acid) (SPANI) films as reported by Nohria *et al.* [28] was 15 s. Qu and Meyer [29] employed the semiconducting metal oxide MnWO_4 to fabricate a thick-film based sensor, which showed an operating range from 25% to 95%RH and response time of 10 s. A humidity sensor of nanostructure Co dispersed in Polyaniline deposited as a clad, having quick response of 8 s (20%–95% RH) and recovery time of 1 min (95% – 20%RH) on fiber optical waveguide, is reported by Vijayan *et al.* [20]. The response and recovery time of the Ag-Polyaniline nanocomposite sensor is given as 7 and 50 s, respectively, which are quite fast.

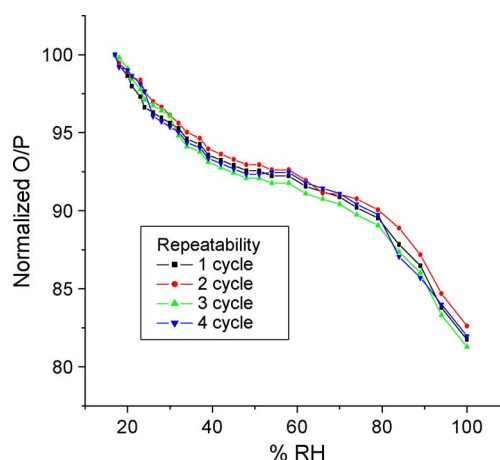


Fig. 9. Cycle-to-cycle variation in sensitivity of Ag-Polyaniline nanocomposite for 27 μm thickness.

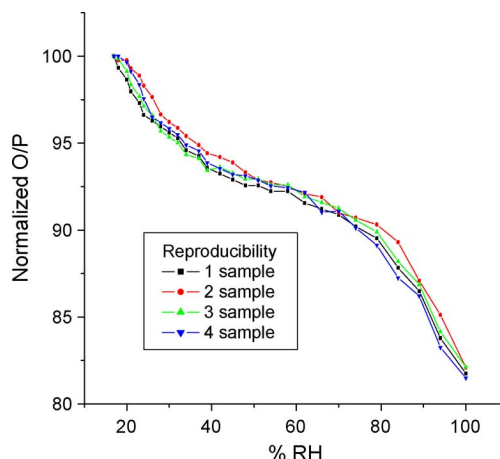


Fig. 10. Sample-to-sample variation in sensitivity of Ag-Polyaniline nanocomposite for 27 μm thickness.

IV. CONCLUSION

A film of Ag-Polyaniline nanocomposite casted by LbL coating is useful for the construction of an optical sensor for determination of relative humidity. It is sensitive in the range of 15%–100% RH. Its behavior is repetitive and the response time of sensor is 7 s with a fast recovery of 50 s, making this sensor useful for RH monitoring in many applications. Laser source can be replaced by simple LED's making it possible to fabricate a simple low-cost sensor.

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