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Fabrication and Calibration of Nanostructured Zno Capacitive Relative Humidity Sensor

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Abstract: *Nanostructured ZnO capacitive relative humidity sensor was successfully designed and fabricated. Tin coated interdigitated electrodes (IDEs) with sensing area of 46.948mm*^{2} *were designed and fabricated to give the sensor its structure.Linear alkylbenzene sulphonate-capped ZnO nanoparticles were used as the sensing material (dielectric material) for the fabrication of the nanostructured ZnO capacitive relative humidity sensor. The nanostructured ZnO RH sensor was fabricated by deposition of the nanostructured ZnO ink on the IDEs using spin coating technique. The relationship between the capacitance values measured by the nanostructured ZnO capacitive RH sensor and the RH measured by DHT11 was determined using some saturatedsalt solutions. The relationship between capacitance and RH variations was 66.85% linear. The RMSE of the sensor was 8.37. The sensitivity of the nanostructured ZnO capacitive RH sensor was 0.161pF/%RH. The sensor was calibrated to obtain 100% linearity using linear regression and a simple model equation. The sensor was successfully calibrated to read relative humidity values ranging from 0%RH to 100%RH.*

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I. Introduction

A sensor detects a change in a physical stimulus and turns it into a signal which can be measured or recorded (Sureshkumar and Rajesh, 2018). The changes in physical quantities could be detected as changes in motion, position, displacement, velocity, acceleration, force, strain, pressure, flow, sound, moisture, light, radiation, temperature and chemical presence (Sinha, 2017). Electronic sensor detects and measures a physical quantity such as humidity, temperature, pressure or loudness and converts it into an electronic signal which could be analog or digital.A sensing system is a complete system that gathers information about events or qualities of the physical world and transforms it into the electronic signals that are used in conventional signal processing systems (Zook, 2008). The microcontroller such as Arduino nano or Arduino uno processes the data from the sensor and sends to the computer system for display of the result. Resistive and capacitive are electronic humidity sensors (Fenner & Zdankiewicz, 2001, Hong *et al*., 2019). The capacitive-type sensor is superior in linearity of sensor output and in stability at high humidity to the resistive-type sensor (Griesel *et al*., 2012). Capacitive humidity sensors have better linearity, accuracy, and higher thermal stability than resistive humidity sensors and sensitivity range of 0.005 pF/% RH to 0.077 pF/% RH (Lee *et al*., 2011).

The performance of a sensor largely depends on its sensing material. Recent studies have attempted to develop more sophisticated humidity sensors by manipulating several sensor characteristics such as refractive index, frequency range, capacitance, impedance and sensing mechanisms (Chen and Liu, 2005; Tripathy *et al*., 2016). These characteristics are strictly determined by the sensing medium material, porosity, surface area and pore size distribution (Tripathy *et al*., 2016). Zinc oxide (ZnO) is one of the most widely applied humidity sensing materials and it has many merits such as its low-cost preparation, plentiful and controllable surface morphology, perfect chemical and thermal stability and high electrical sensitivity to humidity (Chang *et al*., 2010, Yang *et al*., 2017). Zinc oxide (ZnO) is an attractive semiconducting metal oxides owing to its interesting properties including a wide and direct bandgap (3.37 eV) and large exciton binding energy (60 meV) as well as high transparency (Bekkari *et al*., 2017).

This work studies the fabrication of nanostructured ZnO capacitive relative humidity sensor using the interdigitated electrodes and ZnO nanoparticles synthesized by solvothermal technique with linear alkylbebzene sulphonate (LABS) as capping agent.

Design and Fabrication of Interdigitated Electrodes (IDEs) for the Nanostructured ZnO Capacitive Humidity Sensor

Figure 1 represents the schematic of the interdigitated electrodes/sensor's structure and geometrical parameters. The two metallic electrodes form the interdigitated capacitive relative humidity sensor with a comb shape. Each finger of the electrodes has a width W (mm) and a length L (mm). The distance between two consecutive fingers is G (mm) and the total width of the IDEs is H (mm).

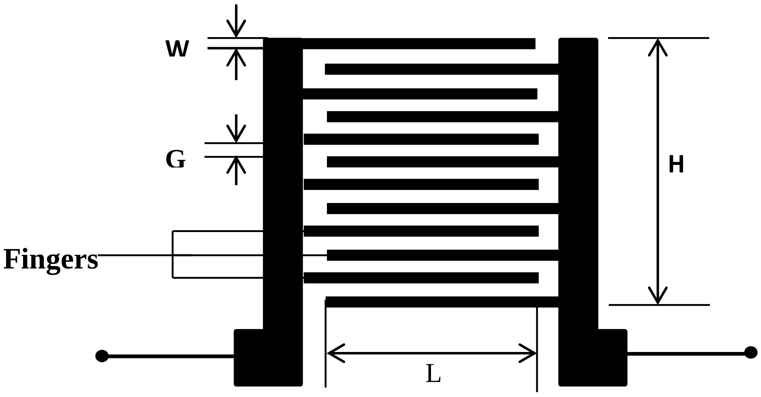

Figure 1 The schematic of the IDEs structure and its parameters

The parameters of IDEs designed for the sensors are as follows:
Number of fingers = 12
Width of a finger (W) = 0.2mm
Length (x-direction) of a finger (L) = 8.536mm
Length (y-direction) of the IDEs (H) = 8.536mm
Spacing/gap between the fingers (G) = 0.5mm

Area of the IDEs = L x H 1
= 8.536 x 8.536
= 72.8633mm^2
The area covered by IDEs gives the area of the sensor.
The area of a finger = L x W 2
= 8.536mm x 0.2mm
= 1.7072mm^2
The total area of the fingers = 12 x 1.7072mm^2
= 20.4864mm^2

The total sensing area (A) of the sensor = 11 (G X L). 3
= 11(8.536 x 0.5)
= 46.948mm^2

The design and fabrication of IDEs involves giving the IDEs a pattern and transferring it ona printed circuit board (PCB). Figure 2 shows the stages used in design and fabrication of the IDEs. The method used in the design was the Generic interdigitated array model. The patterns were designed using a computer aided design program/software (PROTEUS Design Suite). The IDEs designed for the sensor is shown in Figure 2a. The pattern or the design of the IDEs gave the sensor its design. The IDEs were printed with ***hp*** Deskjet F2280 laser printer on a photographic paper. Screen printing (digital printing) was used to transfer the designed IDEs on the PCB.During the screen printing, the print out (the mask) was soaked in HCl acid water for 25 minutes. The photosensitive layer (photoresist) bearing the designed sensor was detached from the photographic paper. The IDE was printed on the PCB using a laminating machine operated at a temperature of 200°C. The etching process was done using a mixture of Ferric Chloride ($FeCl_3$), small amount of HCl and water as the etchant. The Ferric chloride was used because it does not form bubbles or gas. It made the process faster and preserved the features of the design. The PCB was immersed in the ferric chloride mixture for 20 minutes. The IDEprinted on the PCB wasetched and given a copper pattern as shown in figure 2b. Displacement plating/electrochemical plating by immersion tinning was used to coat the copper IDEs with tin (Sn). The IDEs were immersed into the immersion tinning agent of composition of 21gl^{-1} of Tin (II) Chloride, 90gl^{-1} of Thiourea, 3gl^{-1} of benzene disulfonic acid and 0.3M of HCl. In the displacement plating process, as copper ions replaced tin ions in solution, tin metal replaced copper metal on the surface. 5gm of sodium lauryl sulphate was dissolved in 100ml of water and was used in washing the sensor. The sensor was rinsed with distilled water. Propanol was used for the final rinsing to ensure the production of contamination free sensor. The sensor was blown dry using air blower machine. The tin coated IDEs are shown in Figure 2c. Tin coating was to protect the IDEs from corrosion or oxidation and to give it a better contact and make soldering easier. Figure 3 shows a magnified structure of one of interdigitated electrodes after tin coating.

a. IDE design **b. Etched IDE** **b. Tin coated IDE**

Figure 2 Stages in design and fabrication of IDEs

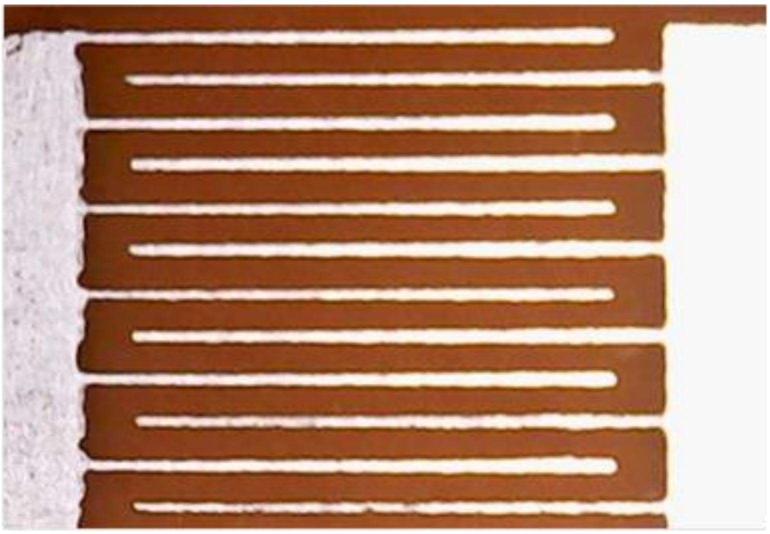

Figure 3 Magnified structures of IDEs after tin coating

Fabrication of the Nanostructured ZnO Capacitive Relative Humidity Sensor

Nanostructured ZnO capacitive relative humidity (RH) sensorwas fabricated using ZnO nanoparticles capped with linear alkylbebzene sulphonate as a dielectric. The nanostructured ZnO ink was prepared by dissolving the powder of the ZnO nanoparticles in propanol. The agglomerate of the nanostructured ZnO ink was sonicated for 30 minutes using Hielscher's ultrasonic sonicator. The sonication was to ensure uniformly dispersed/deagglomerated ZnO nanoparticles. As a result nanostructured ZnO ink with precise particle size and high conductivity was produced.

Spin coating technique was used for the deposition of the nanostructured ZnO ink on the sensors' IDEs. The terminals of the sensor were covered with masking tape to prevent spillage of the ZnO nanoparticles on the terminals during spin coating. The spin coater used for the spin coating was set at the speed of 4000 RPM (revolutions per minute). The IDEs were carefully put in the spin coater. A small puddle of the nanostructured ZnO ink was deposited onto the centre of the IDEs and spun by the centrifugal force for 20 seconds. The IDEs were placed on a temperature controlled hot plate at 150^{0}C for 5 minutes to allow the nanostructured ZnO ink to solidify so that it does not dissolve during the deposition of another layer. The process of the spin coating was repeated four times to obtain five layers of nanostructured ZnO deposited on the IDEs. The fabrication of the nanostructured ZnO capacitive relative humidity sensor was completed. The sensorwasnot annealed. The oxide stain and impurities on the terminals of the sensors were removed using smooth sandpaper. Wires were soldered on the two terminals of the sensoras shown in Figure 4. This is to allow easy connection in an electric circuit.

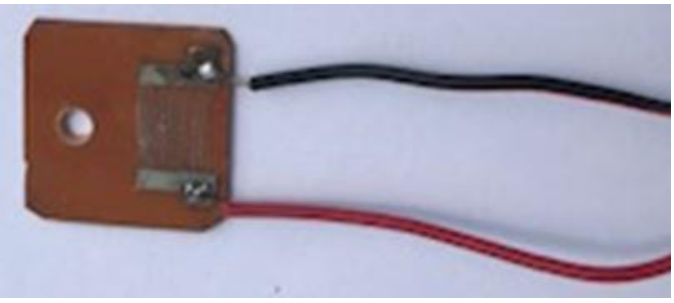

Figure 4 Nanostructured ZnO capacitive RH sensor with wires soldered to the terminals

Relative Humidity Measurement Using the Nanostructured ZnO Capacitive RH Sensor

The ARDUINO NANO ATMEGA 328 microcontroller was programmed to process and measure the capacitance of the nanostructured ZnO capacitive RH sensor and their corresponding humidity values after every second at a specified temperature of the environment. It was programmed to operate at 0V and 5V, where 0V is the digital low (0) and 5V is the digital high (1). Figure 5 shows the ARDUINO Nano ATmega 328 microcontroller operational circuit for the sensor.

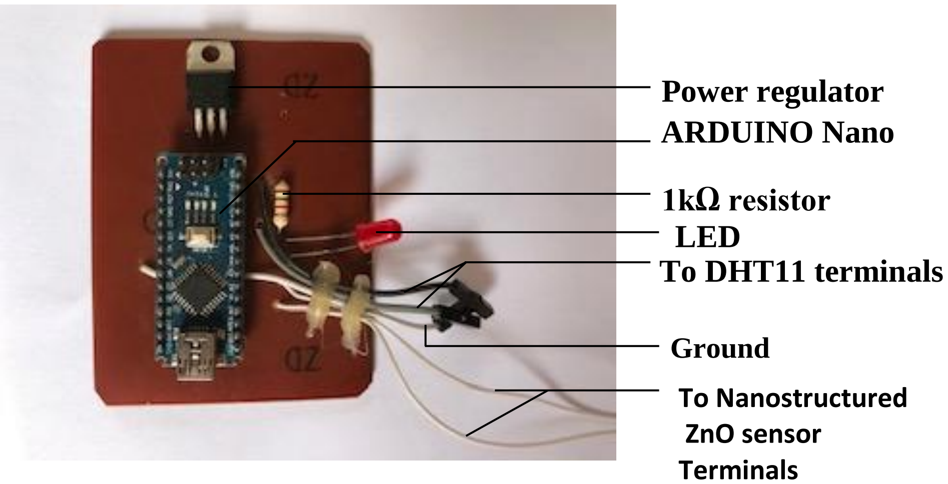

Figure 5 ARDUINO Nano icrocontroller operational circuit

The electronic components used in designing the ARDUINO Nano ATmega 328 microcontroller operational circuit (Figure 5) for the sensor include ARDUNO NANO ATMEGA 328 microcontroller with USB 2.0 – Serial, 1kΩ resistor, light emitting diode (LED) and a voltage regulator provided for external power supply when required. The provisions for connecting to the nanostructured ZnO capacitive relative humidity sensor and DHT11 relative humidity sensor were provided.

Figure 6 shows the ARDUINO Nano ATmega 328 microcontroller electronic humidity sensor system used for the RH measurementscomprised the humidity chamber containing the nanostructured ZnO capacitive relative humidity sensor, DHT11 resistive relative humidity sensor and saturated solution under study; the ARDUINO Nano ATmega 328 microcontroller operational circuit; a lap top computer and a reliable power supply. The capacitance and relative humidity values weremeasured at room (laboratory) temperatureof 28^0C. Stable relative humidity and corresponding capacitance values of lithium Chloride (LiCl), Potassium carbonate (K_2CO_3), potassium chloride (KCl), potassium sulphate (K_2SO_4) and sodium chloride (NaCl) were determined.

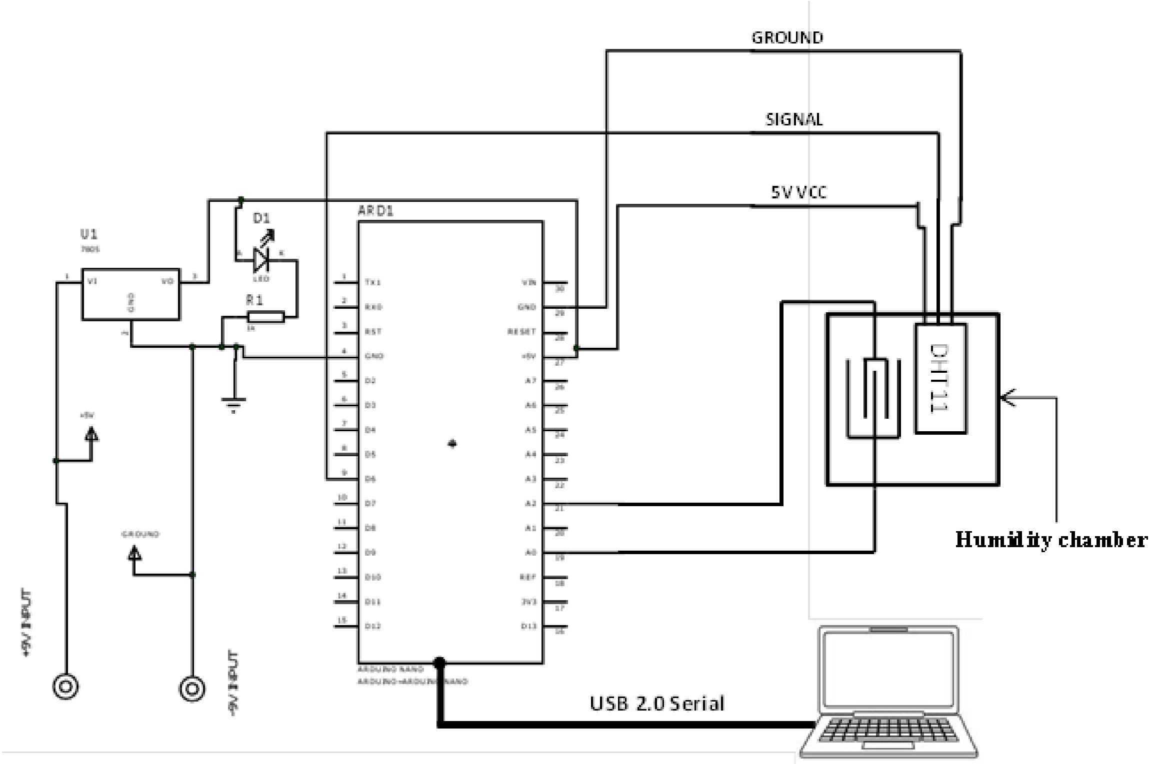

Figure 6 ARDUINO Nano ATmega 328 microcontroller electronic humidity sensor

In each micro humidity environment, the signal from the sensor was sent to the ARDUINO Nano ATmega 328 microcontroller. The data from the sensor was processed by the microcontroller and displayed on the laptop computer. The computer displayed the relative humidity values of the micro humidity environment measured by DHT11 sensor and the corresponding capacitance values measured by the nanostructured ZnO capacitive RH sensor, the temperature detected by the DHT11 sensor and the time for variations for RH and capacitance. The steady (observed) RH value (O%) and the corresponding capacitance (C) were recorded. The sensor's performance was determined using linear regression, root-mean square error (RMSE) and sensitivity of the sensor. The results obtained were used for the calibration of the sensor.

II. Results and Discussions

Table 1 shows the RH values observed by the DHT11 sensor and the corresponding capacitance values observed by the unannealed nanostructured ZnO capacitive RH sensor in the different saturated salt solutions used as micro humidity environments. Figure 7 displays the variations of capacitance with relative humidity. R^2 specified that a linear relationship of 66.85% exists between the capacitance and relative humidity.

Table 1 Capacitance and corresponding RH measured by unannealed nanostructured ZnO capacitive RH sensor in saturated salt solutions (micro humidity environments)

Saturated Salt Solutions	Capacitance (pF)	Relative Humidity (O) %
Lithium Chloride	9.4	13
Potassium Carbonate	11.3	58
Sodium chloride	13.9	86
Potassium Chloride	17.6	87
Potassium Sulphate	22.6	95

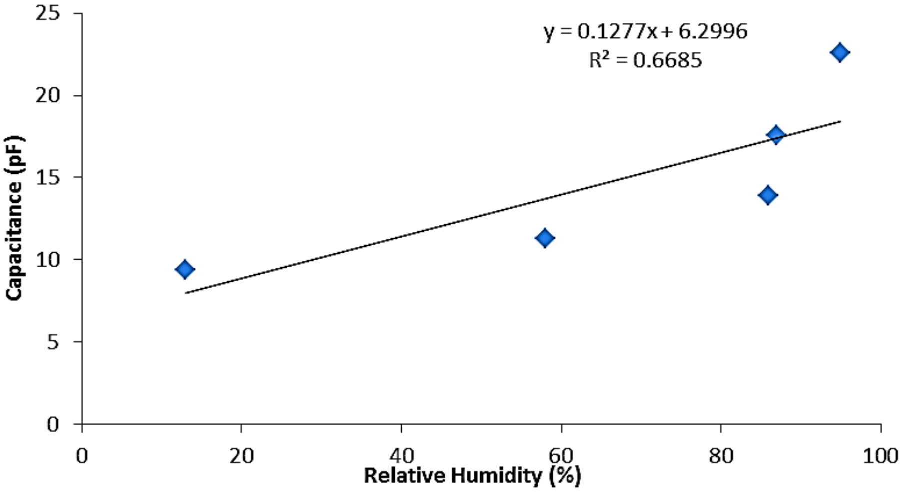

Figure 7 Capacitance versus RH measured by unannealed nanostructured ZnO capacitive RH sensor in LiCl, K_2CO_3, NaCl, KCl and K_2SO_4 in micro humidity environments

The fact that the relationship was not 100% linear necessitatedan error analysis or determination of the accuracy of the sensor and its sensitivity. The root mean square error (RMSE) was used to characterize the sensor's accuracy. The RMSE of the unannealed nanostructured ZnO capacitive RH sensor was calculated using equation 4.

$$\mathrm{RMSE} = \sqrt{\frac{\sum_{i=1}^{n}(O_i - I_i)^2}{n}}, \qquad 4$$

where O_i is the observed sensor value for micro-environment i and I_i is the ideal sensor value for the micro-environment i (i.e., the reference standard). A RMSE of less than 1% implies a high accuracy of the sensor.Table 2compares the reference relative humidity values of the saturated salt solutions and relative humidity values measured by the nanostructured ZnO capacitive relative humidity sensor and the associated errors in measurement. The RMSE was 8.37%. The high RMSE depicted the need for calibration of the sensor.

Table 2 Reference RH and RH measured by nanostructured ZnO capacitive RH sensor and the associated errors

Reference RH		Unannealed Sensor	
Saturated Salt Solution	**I (%RH)**	**O (%RH)**	**Error (%RH)**
Lithium Chloride	11.30	13	1.70
Potassium Carbonate	43.16	58	14.84
Sodium chloride	75.30	86	10.70
Potassium Chloride	84.34	87	2.66
Potassium Sulphate	97.30	95	2.30
RMSE			**8.37**

The sensitivity (S) of the unannealed nanostructured ZnO capacitive relative humidity sensor to relative humidity variations was determined using equations 5.

$$s = \frac{C_H - C_L}{RH_H - RH_L} \qquad 5$$

where S is the sensitivity of the sensor to relative humidity variation, $RH_H$is the highest relative humidity and$RH_L$is the lowest relative humidity measured by the DHT11sensor while C_H is the corresponding highest capacitanceand $C_L$is the corresponding lowest capacitance in the variation range.

$$\Rightarrow S \quad = \frac{22.6-9.4}{95-13} = 0.161\mathrm{pF/\%RH}$$

It implies therefore that the unannealed sensor experienced 0.161pF change in capacitance per 1% change in relative humidity.

The RMSE error in the unannealed nanostructured ZnO capacitive RH sensor was corrected using the sensitivity (S) of the sensor. Since RH is directly proportional to the capacitance of the sensor, an equation (6) relating the reference RH (I), the corresponding capacitance value (C_I) and the sensitivity (S) of the sensor was derived as follows:

$$C_I \propto I$$

$$\Rightarrow C_I = SI \qquad 6$$

where S is the constant of proportionality. Equation 6was used to form table 3 which contains the reference RH of each saturated salt solution and their correct corresponding capacitance values.

Table 3 Reference RH and capacitance for calibration of unannealed nanostructured ZnO capacitive relative humidity sensor

Saturated Slat Solution	**Reference RH (I) %**	**Capacitance (C_I) pF**
Lithium Chloride	11.30	1.81930
Potassium Carbonate	43.16	6.94876
Sodium chloride	75.30	12.12330
Potassium Chloride	84.34	13.57874
Potassium Sulphate	97.30	15.66530

Figure 8 displays the calibration curve of the unannealed nanostructured ZnO capacitive RH sensor. The value of R^2 was 1 which confirmed 100% linearity of the sensor. The slope of the graph was equal to the sensitivity(S) which confirmed it as the constant of proportionality. The intercept was 1.0 x 10^{-14} which implied that the error in the sensor measurement was very negligible.

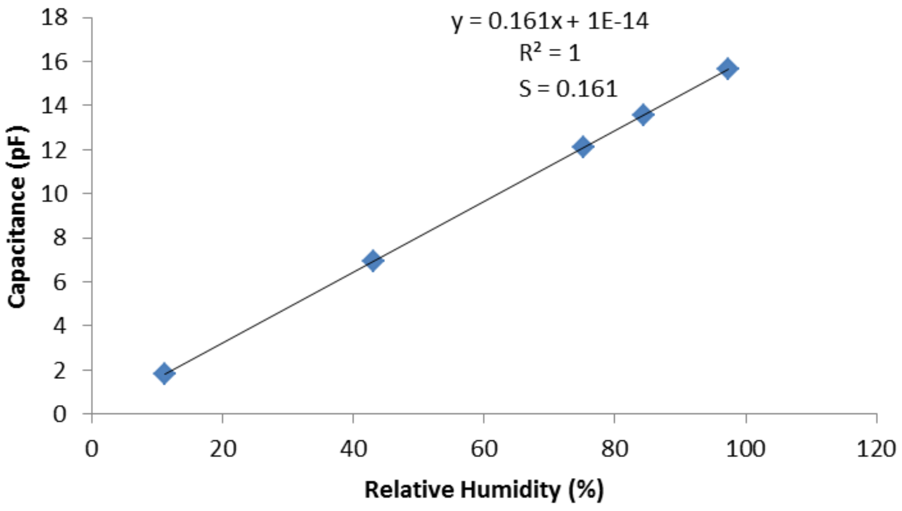

Figure 8 Calibration curve of unannealed nanostructured ZnO capacitive RH sensor

The scale was expanded to read RH values ranging from 0%RH to 100%RH using table 4. The table was developed using arbitrary values of relative humidity and finding their equivalent values using equation 6. The calibration curve for humidity range of 0%RH to 100%RH for unannealed nanostructured ZnO capacitive RH sensor is shown in Figure 9.

Table 4 Expanded scale for the calibration of unannealed nanostructured ZnO capacitive RH sensor

Capacitance (pF)	0	1.61	3.22	4.83	6.44	8.05	9.66	11.27	12.88	14.49	16.10
Relative Humidity (%)	0	10	20	30	40	50	60	70	80	90	100

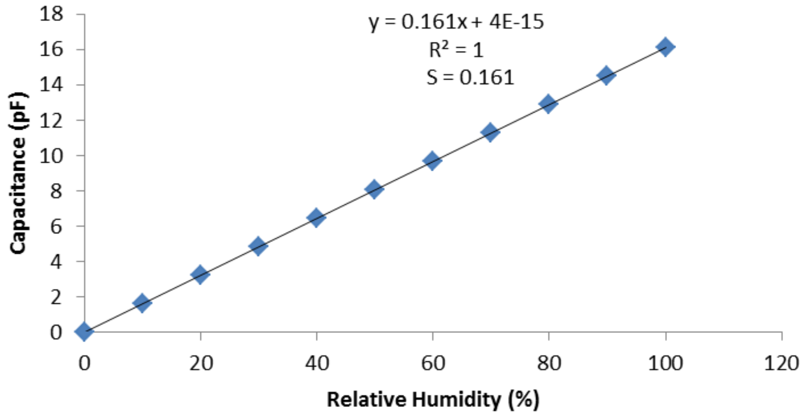

Figure 9 Calibraion curve of unannealed nanostructured ZnO capcitive RH sensor for RH range of 0%RH to 100%RH

III. Conclusion

Interdigitated electrodes (IDEs) with sensing area of 46.948mm^2 were designed. The IDEs were printed on the printed circuit board (PCB) using screen printing (digital printing)technique. ZnO nanoparticles capped with linear alkylbenzene sulphonatewere used as the sensing material (dielectric material) for the fabrication of the nanostructured ZnO capacitive relative humidity sensor. The nanostructured ZnO capacitive relative humidity sensor was successfully fabricated on the IDEs using spin coating deposition technique. The relative humidity measurement from the sensor revealed a linear relationship between the capacitance values measured by the nanostructured ZnO capacitive RH sensor and relative humidity values measured by DHT11 sensor in the saturated salt solutions. The linearity of the sensor's measurement was 66.85%. The root-mean square error of the measurement was 8.37%. The sensitivity of the nanostructured ZnO capacitive RH sensor was 0.161pF/%RH.The sensitivity of the unannealed nanostructured ZnO capacitive relative humidity sensor was improved when compared to 0.005 pF/% RH to 0.077 pF/% RH for capacitive RH sensors reported by Du *et al*. (2004), Lee *et al*. (2011) and Hernández-Rivera *et al*. (2017). It equally shows that ZnO nanoparticles capped with linear alkylbebzene sulphonate is a good sensing material for capacitive RH sensors. The nanostructured ZnO capacitive RH sensor was calibrated to achieve 100% linearity using linear regression and a simple model equation.The sensor was successfully calibrated to read relative humidity values ranging from 0%RH to 100%RH.

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