

In-situ calibration of humidity with simultaneous effect of resistance and capacitance at different frequencies

T. A. Qasuria, S. Alam, S. A. Qasuria, N. A. Qureshi, K. Islam, K. S. Karimov

Abstract— The measurement and control of humidity is one of the most crucial issues in several fields of applications such as automated systems, instrumentation, agriculture, climatology and GIS. Various kinds of materials are being examined for their humidity sensing properties. Here we investigate humidity sensing characteristics of organic-inorganic composite based sensor fabricated on a glass substrate. Carbon nanotubes-zinc oxide (CNTs-ZnO) blend prepared in ethanol is used as organic-inorganic composite layer. The resistance and capacitance of the device are measured as function of relative humidity (RH) at different frequencies. The decrease in resistance and increase in capacitance of the device manifests humidity sensitivity of CNTs-ZnO composite. The capacitive and resistive response of the hygrometer was analyzed at frequencies of 0.1 kHz, 1 kHz and 10 kHz. The sensor shows higher sensitivity at lower frequency as compared with the higher ones.

Index Terms— Relative Humidity, Resistance, Capacitance, Composite film, Frequency.

I. INTRODUCTION

Humidity is important component of our environment and it becomes a vital part of various physical and chemical sensors. The control of humidity is of utmost importance in several industries as well as in daily life. For better sensing humidity sensor must fulfill the following demands; good sensitivity over wide range of humidity, good reproducibility, long life time, no hysteresis, resistance for contaminants, simple structure and low cost. Most of the humidity sensors in modern control system are based on electrical properties such as capacitance, resistance and impedance. A wide range of materials such as polymers, ceramics and composites have been studied in various humidity sensors [1-10]. These polymer electrolytes exhibit good sensing properties and have long stability and reliability compare with other materials. However, these polymers have some shortcomings such as large hysteresis and instability at high humidity [11, 12]. Hence different modifications techniques have been introduced such as cross linking [13, 14], graft polymerization [15-18] and organic-inorganic composite materials [5, 19-23]. Since their discovery in 1993, carbon nanotubes (CNTs) have gained immense interest of scientist throughout the world due

T. A. Qasuria, S. Alam, N. A. Qureshi, K. Islam; Faculty of Engineering Sciences, GIK Institute of Engineering Sciences and Technology, Topi 23640, Khyber Pakhtunkhwa, Pakistan.

K. S. Karimov; Faculty of Electronic Engineering, GIK Institute of Engineering Sciences and Technology, Topi 23640, Khyber Pakhtunkhwa, Pakistan.

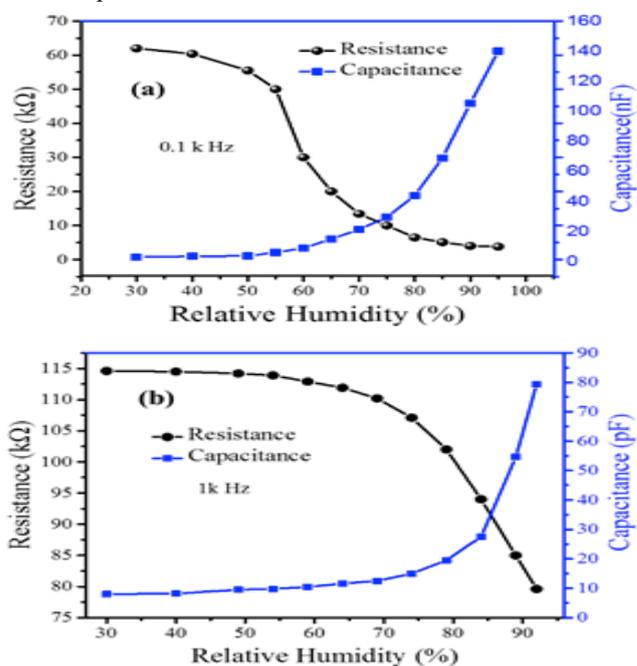
S. A. Qasuria; Department of Chemistry, Gomal University, D.I.Khan, Pakistan

to their remarkable properties in the field of engineering, chemistry, biology and medicine [24-29]. CNTs have been extensively studied for various sensing applications such as humidity, gas and chemical sensing [30-33]. Humidity sensors based on CNTs have shown outstanding sensing properties. Along with CNTs and many other materials, ZnO and its nanostructures are also considered as prominent candidate for humidity sensors. The large surface area of both CNTs and nanostructures of ZnO causes efficient moisture adsorption which produces the change in electrical properties of these materials in diverse humidity environments [34].

In this work, we investigate the humidity sensing properties of organic-inorganic (CNTs-ZnO) composite based hygrometer at different frequencies. CNTs-ZnO composite film as an active layer is deposited on glass substrate with Cu electrodes. The electrical response i.e. resistance, capacitance and impedance of the device are studied as function of RH at 0.1 kHz, 1 kHz and 10 kHz. The decrease in resistance and increase in capacitance with increasing RH at all frequencies manifests the humidity sensitivity of the device.

II. EXPERIMENTAL WORK

The fabrication process of the device is depicted schematically in Fig. 1a. A 1×1 cm² glass substrate was used, which was first washed in deionized water for 15 minutes and then in ultrasonic bath for 30 minutes. Substrate was also plasma cleaned in the.



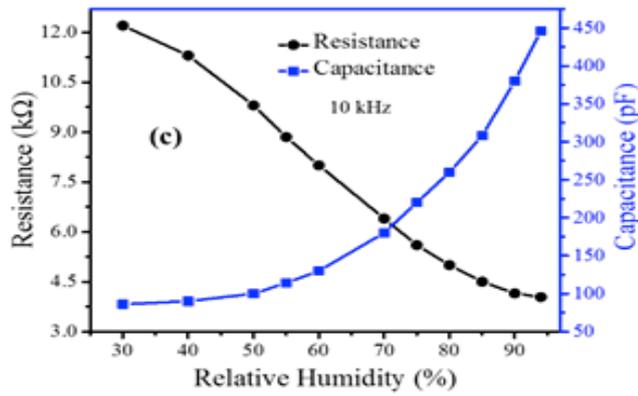


Figure 1: Resistance vs RH (black curves) and capacitance vs RH (blue curves) trends at (a) 0.1 kHz, (b) 1 kHz and (c) 10 kHz. The decrease in resistance and increase in capacitance manifests the humidity sensitivity of the device

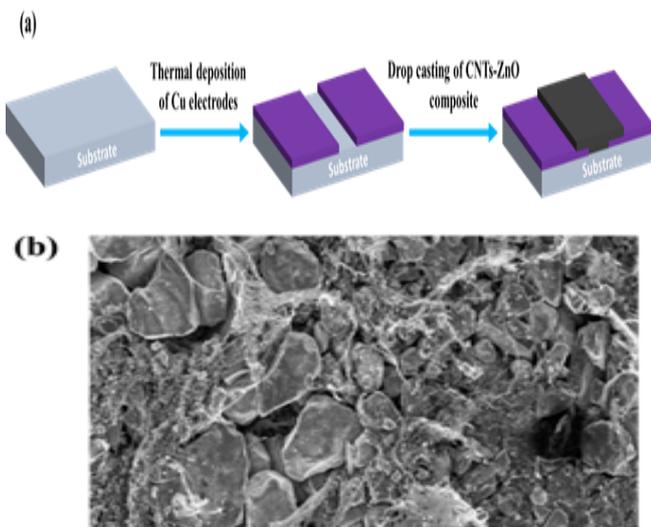


Figure 2: (a) Schematic description of the process used for the fabrication of Cu/CNTs-ZnO/Cu sensor on a glass substrate. (b) SEM image of CNTs-ZnO composite layer.

chamber of thermal evaporator for 5 minutes. The Cu electrodes of 100 nm thickness were deposited on the substrate under high vacuum of 1×10^{-4} mbar at evaporation rate of 0.1 nm/sec by vacuum thermal evaporation technique. A gap of 20 μm was designed between the deposited metal electrodes by using micro-wire mask technique. Humidity sensitive layer i.e. 5wt % solution of CNTs and ZnO prepared in ethanol was drop casted on the substrate. Resistance and capacitance of the device were measured by using a dual display LCR meter (U1732A).

III. RESULTS AND DISCUSSION

The SEM image of the CNTs-ZnO composite film is shown in Fig. 1(b), which reveals the presence of pores in the layer. This porous structure helps in adsorption and condensation water molecules that produces a change in electrical properties. Figure 2(a-c) demonstrates the capacitive and resistive response of sensor with varying RH at 0.1 kHz, 1 kHz and 10 kHz. At 0.1 kHz the resistance decreases sharply from 45-80% RH while the capacitance increases above 50% RH. For the frequency of 1 kHz the change in these

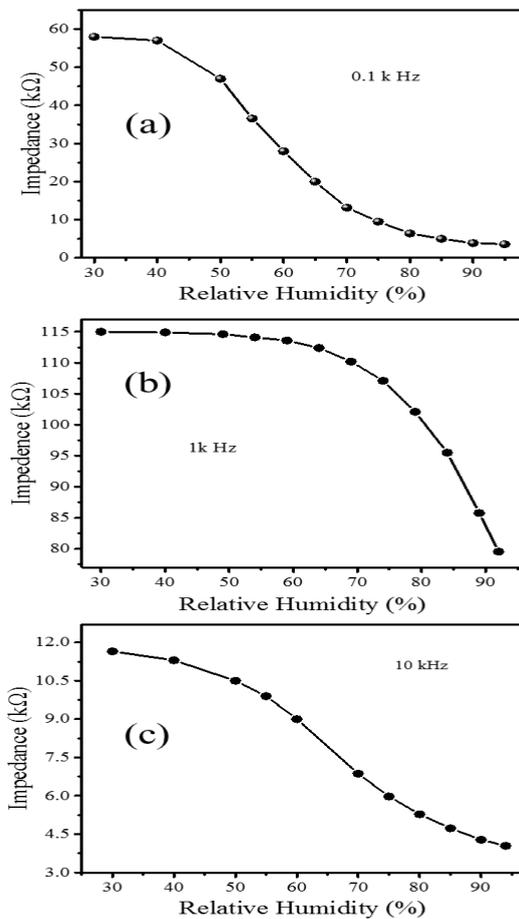


Figure 3: Impedance as function of RH at (a) 0.1 kHz, (b) 1 kHz and (c) 10 kHz

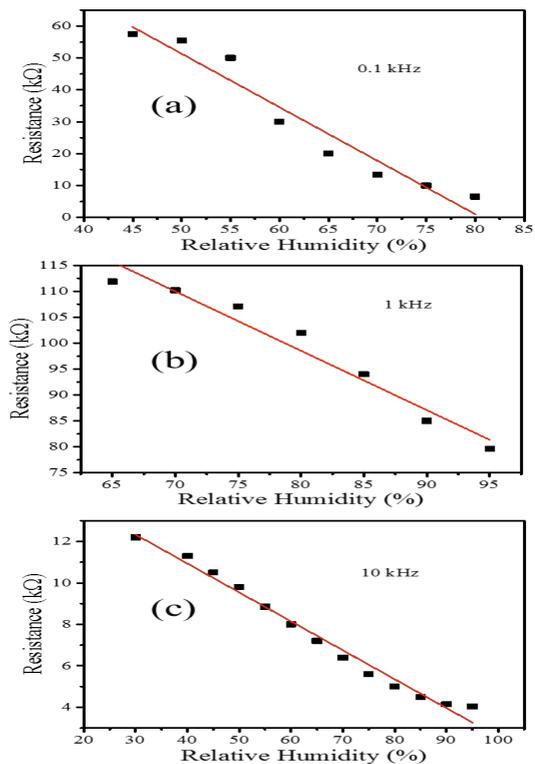


Figure 4: Linear fits of the resistance vs RH curves over a specific range of humidity at the frequencies of; (a) 0.1 kHz, (b) 1 kHz and (c) 10 kHz

electrical parameters is negligible at low humidity and it becomes significant above 60% RH as shown in Fig. 2b. The response of resistance is more linear at 10 KHz over wide range of humidity. The decrease in resistance at all frequencies with increasing RH might be due to displacement current caused by water molecules and the donation of electrons from adsorbed water molecules to surface of semiconducting later [35-37]. In contrast to resistance, the capacitance of the device increases with increase in RH due to enhancement in permittivity of the film caused by adsorption and condensation of water vapors. The capacitance 'C' is given by the relation

$$C = \frac{\epsilon_0 \epsilon_r A}{d} \quad (1)$$

Here, ' ϵ_0 ' is permittivity of space, ' ϵ_r ' is permittivity of dielectric medium, ' A ' is the area of Au plates and ' d ' is separation between them. As the permittivity of water is high so the condensation of water vapors in the pores of active layer causes an increase in the capacitance of the device. The impedance of the sensor also decreases with respect to RH at all three frequencies as demonstrated in Fig. 3(a-c).

To approximate the sensitivity, we have taken the linear fits of resistance vs RH curves in the RH range of 45-80% for 0.1 kHz, 65-95% for 1 kHz and 30-95% for 10 kHz as depicted in Fig. 4(a-c), respectively. The sensitivity 'S' is then estimated by using the relation

$$S = \frac{1}{R_0} \times \frac{\Delta R}{\Delta RH} \times 100 \quad (2)$$

Here, ' R_0 ' is the maximum value of resistance (in Fig. 4 (a-c)), and ' $\Delta R / \Delta RH$ ' is slope of the line. The values of 'S' are estimated to be ~2.8%/RH for 0.1 kHz and ~1%/RH for 1 kHz and 10 kHz.

IV. CONCLUSION

In conclusion, we investigated the humidity sensing characteristics organic-inorganic (CNTs-ZnO) composite based sensor. The device showed good sensitivity over a wide range of humidity. The decrease in resistance and increase in capacitance of the hygrometer with increasing RH manifested the humidity dependence of the electrical response of the device. These measurements were carried out at three different frequencies i.e. 0.1 kHz, 1 kHz and 10 kHz. The sensitivity of the device was observed to be higher at 0.1 kHz as compared with other two frequencies. Our results could be useful for fabrication of low-cost devices for measurement of humidity.

ACKNOWLEDGEMENTS

We wish to acknowledge Faculty of Engineering Sciences and Faculty of Electronic Engineering, GIK Institute for the support extended to this work. We are also thankful to Physical Technical Institute, Tajikistan for their cooperation.

REFERENCES

[1] Park, S., Kang, J., Park, J., Mun, S., One-bodied humidity and temperature sensor having advanced linearity at low and high relative humidity range. *Sensors and Actuators B: Chemical*, 76(1), 322-326, 2001.
[2] Chou, K.-S., Lee, T.-K., Liu, F.-J., Sensing mechanism of a porous ceramic as humidity sensor. *Sensors and Actuators B: Chemical*, 56(1), 106-111, 1999.

[3] Shuk, P., Greenblatt, M., Solid electrolyte film humidity sensor. *Solid State Ionics*, 113, 229-233, 1998.
[4] Ying, J., Wan, C., He, P., Sol-gel processed TiO₂-K₂O-LiZnVO₄ ceramic thin films as innovative humidity sensors. *Sensors and Actuators B: Chemical*, 62(3), 165-170, 2000.
[5] Feng, C.-D., Sun, S.-L., Wang, H., Segre, C.U., Stetter, J.R., Humidity sensing properties of Nation and sol-gel derived SiO₂/Nafion composite thin films. *Sensors and Actuators B: Chemical*, 40(2), 217-222, 1997.
[6] Bernard, M., Humidity sensors. *J. Am. Ceram. Soc.*, 74(4), 697-708, 1991.
[7] Yang, M., Li, Y., Zhan, X., Ling, M., A novel resistive-type humidity sensor based on poly (p-diethynylbenzene). *Journal of applied polymer science*, 74(8), 2010-2015, 1999.
[8] Yang, M., Li, Y., Zhan, X., Sun, H., Ling, M., Zhu, Y., A novel humidity-sensitive material based on PDEB with nickel complex catalyst. *CHINESE CHEMICAL LETTERS*, 9(2), 223-225, 1998.
[9] Sakai, Y., Sadaoka, Y., Matsuguchi, M., Humidity sensors based on polymer thin films. *Sensors and Actuators B: Chemical*, 35(1-3), 85-90, 1996.
[10] Harsányi, G., Polymeric sensing films: new horizons in sensorics? *Materials chemistry and physics*, 43(3), 199-203, 1996.
[11] Kinjo, N., Ohara, S., Sugawara, T., Tsuchitani, S., Humidity sensor with improved protective layering. 1984, Google Patents.
[12] Sharma, Ravindra Kumar, and Sunil Sharma. "Design of HPCF with nearly zero flattened Chromatic Dispersion." *International Journal of Engineering and Applied Sciences*, vol. 1, no. 2, Nov. 2014.
[13] Tsuchitani, S., Sugawara, T., Kinjo, N., Ohara, S., Tsunoda, T., A humidity sensor using ionic copolymer and its application to a humidity-temperature sensor module. *Sensors and Actuators*, 15(4), 375-386, 1988.
[14] Rauen, K.L., Smith, D.A., Heineman, W.R., Johnson, J., Seguin, R., Stoughton, P., Humidity sensor based on conductivity measurements of a poly (dimethyldiallylammonium chloride) polymer film. *Sensors and Actuators B: Chemical*, 17(1), 61-68, 1993.
[15] Sakai, Y., Rao, V., Sadaoka, Y., Matsuguchi, M., Humidity sensor composed of a microporous film of polyethylene-graft-poly-(2-acrylamido-2-methylpropane sulfonate). *Polymer Bulletin*, 18(6), 501-506, 1987.
[16] Sakai, Y., Sadaoka, Y., Humidity sensors using sulfonated microporous polyethylene films. 1985, ELECTROCHEMICAL SOC JAPAN 12-1 I CHOME YURAKU-CHO, CHIYODA-KU, TOKYO 100, JAPAN. p. 150-151.
[17] Sakai, Y., Sadaoka, Y., Matsuguchi, M., Rao, V., Humidity sensor using microporous film of polyethylene-graft-poly-(2-hydroxy-3-methacryloxypropyl trimethyl-ammonium chloride). *Journal of materials science*, 24(1), 101-104, 1989.
[18] Sakai, Y., Sadaoka, Y., Matsuguchi, M., Rao, V., Kamigaki, M., A humidity sensor using graft copolymer with polyelectrolyte branches. *Polymer*, 30(6), 1068-1071, 1989.
[19] Wang, J., Xu, B., Zhang, J., Liu, G., Zhang, T., Qiu, F., Zhao, M., Humidity sensors of composite material of nanocrystal BaTiO₃ and polymer (R) nM⁺ X⁻. *Journal of materials science letters*, 18(19), 1603-1605, 1999.
[20] Wang, J., Lin, Q., Zhou, R., Xu, B., Humidity sensors based on composite material of nano-BaTiO₃ and polymer RMX. *Sensors and Actuators B: Chemical*, 81(2), 248-253, 2002.
[21] Sharma, Ravindra Kumar, et al. "A design of hybrid elliptical air hole ring chalcogenide As₂Se₃ glass PCF: application to lower zero dispersion." *International Journal of Engineering Research and Technology*, vol. 1, no. 3, May 2012.
[22] Su, P.-G., Tsai, W.-Y., Humidity sensing and electrical properties of a composite material of nano-sized SiO₂ and poly (2-acrylamido-2-methylpropane sulfonate). *Sensors and Actuators B: Chemical*, 100(3), 417-422, 2004.
[23] Li, Y., Yang, M., She, Y., Humidity sensors using in situ synthesized sodium polystyrenesulfonate/ZnO nanocomposites. *Talanta*, 62(4), 707-712, 2004.
[24] Iijima, S., Ichihashi, T., Single-shell carbon nanotubes of 1-nm diameter. *Nature*, 363(6430), 603-605, 1993.
[25] Yakobson, B.I., Smalley, R.E., Fullerene nanotubes: C 1,000,000 and beyond: Some unusual new molecules—long, hollow fibers with tantalizing electronic and mechanical properties—have joined diamonds and graphite in the carbon family. *American Scientist*, 85(4), 324-337, 1997.

- [26] Davis, J.J., Coleman, K.S., Azamian, B.R., Bagshaw, C.B., Green, M.L., Chemical and biochemical sensing with modified single walled carbon nanotubes. *Chemistry-a European Journal*, 9(16), 3732-3739, 2003.
- [27] Wong, E.W., Sheehan, P.E., Lieber, C.M., Nanobeam mechanics: elasticity, strength, and toughness of nanorods and nanotubes. *science*, 277(5334), 1971-1975, 1997.
- [28] Dekker, C., Tans, S., Devoret, M., Dai, H., Smalley, R.E., Thess, A., Georlga, L., Individual single-wall carbon nanotubes as quantum wires. *Nature* 386 (6624), 474-477.(1997), 1997.
- [29] Hone, J., Batlogg, B., Benes, Z., Johnson, A., Fischer, J., Quantized phonon spectrum of single-wall carbon nanotubes. *Science*, 289(5485), 1730-1733, 2000.
- [30] Yoo, K.-P., Lim, L.-T., Min, N.-K., Lee, M.J., Lee, C.J., Park, C.-W., Novel resistive-type humidity sensor based on multiwall carbon nanotube/polyimide composite films. *Sensors and Actuators B: Chemical*, 145(1), 120-125, 2010.
- [31] Zhang, T., Mubeen, S., Myung, N.V., Deshusses, M.A., Recent progress in carbon nanotube-based gas sensors. *Nanotechnology*, 19(33), 332001, 2008.
- [32] Chen, W.-P., Zhao, Z.-G., Liu, X.-W., Zhang, Z.-X., Suo, C.-G., A capacitive humidity sensor based on multi-wall carbon nanotubes (MWCNTs). *Sensors*, 9(9), 7431-7444, 2009.
- [33] Sharma, Ravindra Kumar, Kirti Vyas, et al. Investigation of Zero Chromatic Dispersion in Square Lattice As₂Se₃ Chalcogenide Glass PCF. Jan. 2012.
- [34] Zhang, Y., Yu, K., Jiang, D., Zhu, Z., Geng, H., Luo, L., Zinc oxide nanorod and nanowire for humidity sensor. *Applied Surface Science*, 242(1), 212-217, 2005.
- [35] Karimov, K.S., Saleem, M., Qasuria, T., Farooq, M., Surface-type humidity sensor based on cellulose-PEPC for telemetry systems. *Journal of Semiconductors*, 32(1), 015005, 2011.
- [36] Boyle, J., Jones, K., The effects of CO, water vapor and surface temperature on the conductivity of a SnO₂ gas sensor. *Journal of Electronic Materials*, 6(6), 717-733, 1977.
- [37] Chen, Z., Lu, C., Humidity sensors: a review of materials and mechanisms. *Sensor letters*, 3(4), 274-295, 2005.