Humidity and temperature sensing properties of 2-{(Z)-(1-(ethoxycarbonyl)-2-oxopropyl)diazenyl}benzoic acid

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Abstract
In this paper, surface type 2-{(Z)-(1-(ethoxycarbonyl)-2-oxopropyl)diazenyl}benzoic acid (Z-ECODBA) based humidity and temperature sensors have been fabricated and investigated. Thin films of aluminum electrodes and organic semiconductor (Z-ECODBA) were thermally deposited onto the surface of a cleaned glass substrate using Edward Auto 306 thermal evaporator. The surface morphology of the active thin film of Z-ECODBA was studied using Scanning Electron Microscopy (SEM). The humidity dependent capacitive and resistive responses of Z-ECODBA based sensors were studied in the relative humidity range of 40% RH to 90% RH at different applied frequencies. The results showed that the capacitance increased nonlinearly with the increasing relative humidity level, while the resistance decreased and vice versa. The value of capacitance increased 1551.43, 628.18, 376.96, 119 and 71 times for sample (A), 848.75, 350, 175, 112.5 and 50 times for sample (B), 691.45, 275, 177.34, 69.87 and 48.6 times for the sample (C) at 0.1, 0.5, 1, 5 and 10 kHz, respectively. The decrease in resistance was observed 26712.09, 1281.11, 1130, 578.60 and 173.41 times for sample (A), 23327.29, 1992.307, 7047.36, 22455.55 and 12400 times for the sample (B) and 29435.72, 5341.86, 3732.25, 2642.10 and 1495 times for sample (C) at a frequency of 0.1, 0.5, 1, 5 and 10 kHz, respectively. The effect of temperature on the capacitance of the fabricated samples was investigated at three different applied frequencies of 0.1 kHz, 0.5 kHz and 1 kHz, respectively. The capacitance has also been observed to increase with the rise in temperature. Moreover, the capacitance was found to decrease while resistance was observed to increase with the increasing inter electrodes gap. The response and recovery times were recorded 30 s and 21 s for sample of 25 μm, 27 s and 21 s for sample of 40 μm and 27 s and 27 s for sample of 50 μm inter electrodes gap, respectively.

1. Introduction
In the last few decades, extensive research work has been carried out using organic materials in thin film electronic and optoelectronic devices due to their interesting electrical, mechanical and optical properties [1–3]. These materials have been employed in various type of thin film electronic and optoelectronic devices such as organic solar cells, light emitting diodes, schottky diodes, sensors, transistors, photo detectors, etc [4–10]. The sensitivity of these material to the ambient condition makes them suitable for the development of humidity, temperature, light, pressure and gas sensors [11–15]. These sensors have significant applications in our daily life, such as in medicine storage, fresh and packaged food, for safety purposes in laboratories and industries etc [16–19].

The sensors based on organic semiconductor can be fabricated in two type of geometry such as surface type and sandwich type structures. In sandwich type structure, the top electrode may damage thin film of active organic material which results in device failure problem [20, 21]. Therefore, research interest has been given to the fabrication of surface type thin film devices [22–25].

Compared to polymers, thin films of organic dye materials pose many advantages such as long term stability, reproducibility and long range sensitivity [26, 27]. Therefore, research attention has been recently paid to dye...
materials in sensors technology. The researchers are continuously synthesizing and investigating new organic dyes to manipulate the advantages of these materials for the development of different types of sensors. The application of organic dye using (E)-2-{4-[2-(3,4,5-trimethoxybenzylidene)hydrazinyl]phenyl}ethylene-1,1,2-tricarbonitrile (TMBHPET) and aluminum electrodes in thin film capacitive humidity sensor was investigated by Al-Sheme et al. The high sensitivity to humidity with good linearity and fast response and recovery times were recorded. The study of (E)-2-(4-(2-(3,4-dimethoxybenzylidene)hydrazinyl)phenyl)ethane-1,1,2-tricarbonitrile (DMBHPET) organic dye in thin film surface type humidity sensors have been investigated by Azmer et al. The capacitance and resistance of the devices were found to be dependent upon the relative humidity level.

This paper reports surface type humidity and temperature sensors, using newly synthesized organic dye (Z-ECODBA) as an active sensing material. Three devices of various inter electrodes gap i.e., 25 μm, 40 μm and 50 μm named as sample A, sample B and sample C have been fabricated. The effects of humidity and temperature on the capacitance and resistance of the fabricated surface type sensors have been examined at various applied frequencies. The response and recovery times for the fabricated samples have also been studied.

2. Experimental procedure

2.1. Synthesis of 2-{(Z)-[1-(ethoxycarbonyl)-2-oxopropyl]diazenyl} benzoic acid

Anthranilic acid (1g, 0.00724mol) was dissolved in water by adding 3 ml HCl and the mixture was kept cool just below 0 °C in ice bath, followed by drop wise addition of aqueous NaNO2 solution with continuous stirring for 20 min to ensure complete diazotization. To the diazotized solution was then added ethylacetoacetate dissolved in ethanol containing catalytic amount of sodium acetate and the combined mixture was stirred at 0 °C for 20 min followed by continuous stirring for the next 30 min at room temperature. The product was precipitated as yellow green color precipitate and was recrystallized from water-ethanol mixture to get pure product. Yield: 92.92%, m.pt. 138 °C in ice bath, followed by drop wise addition of aqueous NaNO2 solution with continuous stirring for 20 min to ensure complete diazotization. To the diazotized solution was then added ethylacetoacetate dissolved in ethanol containing catalytic amount of sodium acetate and the combined mixture was stirred at 0 °C for 20 min followed by continuous stirring for the next 30 min at room temperature. The product was precipitated as yellow green color precipitate and was filtered. The precipitate so obtained was washed with water and recrystallized from water-ethanol mixture to get pure product. Yield: 92.92%, m.pt. 138 °C–140 °C. Figure 1 shows the molecular structure of 2-{(Z)-[1-(ethoxycarbonyl)-2-oxopropyl]diazenyl} benzoic acid.

2.2. Device fabrication

Initially glass substrates were cleaned ultrasonically with acetone and then rinse with deionized water and dried in an open atmosphere. Thin films of thickness (100 nm) of aluminum were thermally deposited on the surface of cleaned glass substrates using a thermal evaporator (Edwards Auto 306). The gaps of 25 μm, 40 μm and 50 μm were made between the metallic electrodes using a proper mask. The deposition rate and thickness of the film were monitored by FTM5 crystal-controlled thickness monitor. Thin layers of organic material of thickness 100 nm were thermally deposited in the gaps between the metallic electrodes using Edwards Auto 306 thermal evaporator. The pressure was kept 10⁻⁵ mbar during the process of deposition. The surface type humidity and temperature sensors of structure Al/Z-ECODBA/Al were fabricated and shown in figure 2. The humidity dependent capacitive and resistive measurements were carried in a self-made humidity chamber, where the humidity was controlled by the injection of nitrogen (N2) gas after passing through the water chamber. Digital hygrometer (RH 101) was used to measure humidity level and LCR meter (GW Instek 817) was used to observe capacitance-relative humidity and resistance-relative humidity relationships at different applied frequencies. The temperature dependent measurements were made by placing the sample in a chamber which is heated by an electrical heater. The temperature-capacitance relationships at different applied frequencies were observed by using LCR meter (GW Instek 817).
3. Results and discussion

The surface morphology of the thermally deposited thin film of organic material was analyzed using SEM at a different magnification as shown in figure 3. The SEM micrographs show that the deposited film is porous and rough. Also at high magnification micrographs platelets and rod shaped regions can be observed in different sizes and orientations, which also play a significant role in the absorption of moisture. These platelets and rod shape morphology of the grains formed in the sample are due to temperature evaporation during deposition via thermal evaporator. The porosity plays a significant role in the fabrication of humidity sensor because the response of humidity sensor is greatly dependent on the amount of water vapors absorbed within the pores and also by the condensation of the water in the pores of the deposited thin film [30].

Figure 4 shows the capacitance-relative humidity relationships of Al/Z-ECODBA/Al surface type sensors at different applied frequencies. The experimentally obtained graphs show that the value of capacitance increases
nonlinearly with the increasing relative humidity level. The increase in capacitance of the sample (A) shown in figure 4(A) against RH were observed 1551.42, 628.18, 388.75, 122.90 and 104.33 times at a frequency of 100 Hz, 500 Hz, 1 kHz, 5 kHz and 10 kHz, respectively. However, the increase in capacitance were found 848.75, 350, 175, 112 and 50 times for the sample (B) shown in figure 4(B) and 691.44, 275, 177.34, 69.87 and 48.67 times for the sample (C) shown in figure 4(C), at a frequency of 100 Hz, 500 Hz, 1 kHz, 5 kHz and 10 kHz, respectively. These results can be explained on the basis of absorption of H₂O molecules within the pores of active thin film which increases the dielectric constant and as a result the increase in capacitance was observed. Generally, the dielectric constant of the water is higher i.e., 80, but that of organic semiconductors is low, i.e., lying in the range of 4 to 8. So the absorption of water molecules increases the dielectric constant of the active thin film [31]. The amount of absorbed water vapors depends on the porosity of the thin film and the relative humidity level [32]. Also, due to the polar nature of water molecules their dipole moment is strongly affected by the electric field. The dipole moment and electric field are related by the following expression:

\[ P = \alpha E \]

where \( \alpha \) is the polarizability and E is the electric field. There are basically three sources of polarizability such as ionic, dipolar and electronic, which play role in the enhancement of dipole moment. With the absorption of water molecules, the dipolar polarizability increases, which also increase the dipole moment and consequently the capacitance increases. It is also observed that the effect of humidity is less in low humidity range while it becomes more effective at higher humidity level. This is due to the fact that the concentration of absorbed water within the pores of the thin film increases at higher humidity level.

In order to test the sensitivity of the fabricated samples \( \Delta C/C_0 \) against RH at different operating frequencies are plotted in the figure 5, Where

\[ \Delta C = C_H - C_0 \]

\( C_H \) is the capacitance at any relative humidity and \( C_0 \) is the initial capacitance.

It is inferred from the graph that the fabricated sensor is more sensitive to lower frequencies. The sensitivity of the sample (A) is 1550.42 at the lowest applied frequency of 100 Hz and 70 at the highest applied frequency of
Figure 5. Represent the sensitivity of the fabricated samples.

Figure 6. Resistance-relative humidity graphs of the sample (a) 25 μm (b) 40 μm (c) 50 μm inter electrodes gap.
10 kHz, respectively. However, the sensitivity is 678 and 49 for sample (B) and 690 and 47.67 were observed for the sample (C) at the lowest frequency of 100 Hz and highest frequency of 10 kHz, respectively. Therefore, 100 Hz is the best applied frequency for the fabricated sensor. It can also be observed that the response of the fabricated sensor is more prominent after 70 RH may be due to the change in the absorption mode from the monolayer chemisorption to multilayer physisorption at low and high relative humidity [33].

Figure 6 presents the variation of resistance against the increasing relative humidity level observed at different applied frequencies. The obtained curves show the decrease in resistance against the increasing relative humidity level in the range of 40% RH to 90% RH. The decrease in resistance against the increasing relative humidity level for the sample (A) were observed 26712.09, 1281.11, 1130, 578.60 and 173.41 times at a frequency of 100 Hz, 500 Hz, 1 kHz, 5 kHz and 10 kHz, respectively (figure 6(A)). Where, the change in resistance were found 23327.29, 1992.307, 7047.36, 22455.55 and 12400 times for the sample (B) and 29435.72, 5341.86, 3732.25, 1495 times for sample (C) at a frequency of 100 Hz, 500 Hz, 1 kHz, 5 kHz and 10 kHz, respectively. The large decrease in resistance prove that the absorption of water molecules has a great influence on the conductivity of the active sensing film. The decrease in resistance may be due to dissociation of absorbed water molecules into ionic functional groups such as H⁺, OH⁻ which increase the electrical conductivity. The conductivity may also be increased with the absorption of polar water molecules due to the increase of proton concentration in the active thin film [12].

Figure 7 represents the resistive sensitivity of the fabricated sensor at different applied frequencies. It can be deduced from the response of the fabricated sensor that it is more sensitive in the lower frequency and low humidity range. The response signal shows good linearity for the given relative humidity range.

Figure 8 shows the capacitance-frequency relationships observed at different RH levels. It should be noticed that the value of capacitance decreases with increasing the applied frequency and the rate of decrease is more prominent at higher relative humidity. However, generally the capacitance of ideal capacitive sensor is independent of the applied frequency. But due to the absorption of water molecules the active sensing film possess a leak conduction (γ) which is related to the capacitance by the expression [33]
where $\varepsilon^*$ is the complex dielectric constant, $C_0$ is the capacitance of ideal capacitor, $\varepsilon_r$ is the relative dielectric constant, $\omega$ is the angular frequency, $\gamma$ is the conductance and $\varepsilon_0$ is the permittivity of the free space. It should be observed from equation (2) that the capacitance of the fabricated samples is directly proportional to $\gamma$ and inversely proportional to the frequency ($\omega$). Thus, increasing frequency decreases $C$ value. Furthermore, increasing RH at constant $\omega$ increases $\gamma$ and as a result capacitance value increases.

Figure 9 shows the dependence of resistance on the applied frequency of the samples observed at different percentage of relative humidity levels. The obtained results showed that the resistance of the samples decreased with the increasing applied frequency. This phenomenon may be due to the insufficient relaxation time of the charge carrier at the higher applied frequency. However, at low applied frequency the slowly changing polarity of electric filed resulting the space charge polarization phenomenon [34].

Figures 10(A)–(C) represents the capacitance-inter electrodes gap relationships of Al/Z-ECODBA/Al surface type sensor. The experimentally obtained results present the decrease in capacitance with the increase of the gap between metallic electrodes. The results in figure 10 can be explained by considering the edge of the deposited metallic electrodes as parallel plate capacitor. Using this approximation than the capacitance and inter electrodes gap are related by the expression [29]

$$C = \varepsilon_r \varepsilon_0 \frac{A}{d}$$

where $\varepsilon_0$ is the permittivity of free space, $\varepsilon_r$ is the permittivity of the materials, $A$ is the area of one of the plate of capacitor and $d$ is the separation between plates, respectively. The results obtained in figure 10 shows the validity of equation (3).

The resistance-inter electrodes gap relationships at a relative humidity level of 50 RH is presented in figures 11(A)–(C). It is observed that the resistance of the Al/Z-ECODBA/Al humidity and temperature sensor...
Figure 9. Resistance-frequency plots of the fabricated sample (A) sample (B) and sample (C).

Figure 10. (A)–(C). Capacitance-inter electrodes gap relationships of the fabricated samples at two different applied frequencies.
increases with the increasing gap between electrodes. This might be due to the inverse relation between capacitance and resistance expressed as

\[
R = \frac{1}{2\pi f CD}
\]

(4)

where \(R\) is the resistance, \(f\) is the applied frequency, \(C\) is the capacitance and \(D\) is the dissipation constant. It is inferred from equations (3) and (4) that increasing inter electrodes gap decrease capacitance and consequently increasing resistance. Thus the results in figure 11 is the validity of equation (3).

The response and recovery times for Z-ECODBA based sensor have been investigated to observe the performance of the fabricated samples. The response time is defined as the time taken by the sensor to absorb water molecules when the humidity level is suddenly increased from 40% RH to 90% RH. Similarly, the recovery time is the time taken by the sensor to recover itself when the relative humidity has abruptly decreased from 90% RH to 40% RH. The response time was measured by abruptly increasing the humidity from 40% RH to 90% RH while, the recovery time was measured by rapidly decreasing humidity from 90% RH to 40% RH. The response and recovery times were observed 30 s and 21 s for sample (A), 27 s and 21 s for sample (B) and 27 s and 27 s for sample (C) shown in figures 12(A)–(C). Repeated cycles between these two humidity levels give us almost similar curves.

The nonlinear responses on relative humidity dependent capacitive measurement may be a drawback for the standardization of the practical application of the fabricated humidity and temperature sensor. Therefore, an exponential function can be introduced to make such nonlinear behavior more linear. Thus, the logarithmic capacitance-relative humidity curves were generated and shown in figures 13(A)–(C). The two different slop linear relationship in log\(C\)-RH curve intersecting at 60% RH were observed in figures 13(A)–(C). The intersecting point called critical point may indicate the switching of the absorption mode of water molecules from monolayer called chemisorption to multilayer called physisorption [35]. The experimentally observed data points were found to be well fitted linearly with a slope of 0.05 and intercept \(-13.41\) in relative humidity range of 40%–60% RH, and with intercept and slope of \((-14.57, 0.0706)\) were found in the higher humidity range of 60%–90% RH for the sample (A) shown in figure 13(A). The regression \((R^2)\) value of both the fitted curves for
Figure 12. Response and recovery time graph of the fabricated samples.

Figure 13. (A)–(C). The logC–RH curve of the sample (A), (B) and (C) at applied frequency of 100 Hz.
sample (A) were found to greater than 0.97, representing the best fit to improve linearity. Similarly, the increase in slop after 60 %RH were observed for the sample (B) and sample (C) shown in figures 13(B)–(C). However, the R² were found 0.91 and 0.97 for sample (B) and were observed 0.81 and 0.97 for sample (C). The linearity consideration of logarithmic curve against relative humidity represents the breakthrough in the field of organic semiconductor based sensors.

Figure 14 presents the capacitance versus temperature characteristic of the Al/Z-ECODBA/Al surface type humidity and temperature sensors. These characterizations were made under dark conditions with constant relative humidity of 40% RH. The graphs show that the value of capacitance increases nonlinearly with the increase in temperature from 15 °C- 100 °C. The capacitances of the sample (A) were found to increase 10.5, 9.13, 6.52 time at a frequency of 100 Hz, 500 Hz and 1 kHz, respectively. Similarly, the increase in capacitance of the sample (B) were recorded 10, 8.22 and 5.21 time while for sample (C) the changes were found 11.38, 6.78 and 2.76 time at applied frequency of 0.1 kHz, 0.5 kHz and 1 kHz, respectively. The polarization and conductance of the active sensing materials may cause the enhancement of capacitance of the sensor [33]. As the temperature increases, the thermal motions of the molecules become stronger, which increases the polarization and causing the increase in output capacitance.

4. Conclusion

In the present research work, surface type temperature and humidity sensors have been fabricated and investigated using 2- {[(Z)-1-(ethoxycarbonyl)−2-oxopropyl]diazeyln} benzoic acid. The capacitance of the fabricated samples increased while the resistance was found to decrease with the increase in relative humidity level. Moreover, both the capacitance and resistance were observed to decrease with the increasing applied frequency. The increase in capacitance against relative humidity of the sample A were observed 1551.42, 628.18, 388.75, 122.90 and 104.33 times, the increase in capacitance of sample B were found 848.75, 350, 175, 112 and 50 times and 691.44, 275, 177.34, 69.87 and 48.67 times for the sample C, at a frequency of 100 Hz, 500 Hz,
1 kHz, 5 kHz and 10 kHz, respectively. The temperature dependent measurements showed that the capacitance of the fabricated devices increases with the rise in temperature.

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