

Nanoscopic Polyindole DNA-Templated Gas Sensors for Volatile Organic Compounds

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Abstract

In recent times, there has been an increasing demand for inexpensive, accurate, movable and reliable gas sensors that can differentiate between very low concentrations of analytes. Conductive polymers (CPs) such as indole are being explored as promising materials for micro sensors, because of their ability to form a good basis for premise chemical sensors either as a sensing element or as a matrix to deactivate specific analytes. In the previous work the chemical, morphological and electrical characterisation of the Polyindole/Deoxynucleic Acid (PIn/DNA) nanowires have shown the formation of a smooth hybrid material with good electrical conductivity. In this research work, the development of a gas sensor based on PIn/DNA nanowires for the detection of volatile organic compounds (VOCs) at room temperature was reported. For the response time, chloroform (104 Secs) takes the longest time to respond when exposed to the PIn/DNA bulk nanowires while ethanol and hexane both have responses time of less than 80 Secs. While for the recovery time, ethanol (65 Secs) was recovered faster than all the other VOCs while acetone (93 Secs), chloroform (98 Secs), and methanol (91 Secs). Overall, response time is very fast in comparison with the recovery time. Measurement of response time revealed ethanol to have the highest ratio (1.0 Secs) and hexane (0.71 Secs) have the least values. On the other hand, the highest baseline drift value was observed in chloroform (1.5 Secs) while the least of 1.0 Secs was recorded for propanol. Finally, for the sensitivity, Hexane (0.58) has the lowest towards the PIn/DNA nanowires, while methanol has the highest (1.3). The result suggests the possibility of using the PIn/DNA sensor for measuring VOCs at room temperature.

Keywords: sensing, polymer, selectivity, temperature analytes

Introduction

Chemical research has focused on low dimensional nanomaterials due to their unique properties superior to those of the bulk material that can be attributed to their surface and macroscopic quantum tunnelling effects. Highly developed synthetic procedures, with carefully controlled solid state properties, in inorganic semiconductor and metal nanomaterials, have resulted in a rich array of nanoscale devices with superior properties to those of conventional microelectronics and optoelectronics. Recently, a lot of nanomaterials have been explored for their applications in sensors for environmental monitoring of hazardous substances, making their applications a fast-evolving field. Nanomaterials introduce versatility to the sensing platforms and may even allow mobility between different detection mechanisms. The prospect of a combination of different nanomaterials allows an exploitation of their synergistic additive and novel properties for sensor development. (Steindl and Klenovský, 2022; Xu and Lin, 2020; Gao et al., 2019).

Conductive polymers (CPs) are repeated sequences of monomer units that exhibit sp^2 hybridised carbon atoms. The π -conjugated structure with alternating double/single bonds gives rise to their band gap state; addition of dopants, typically anions, makes pristine CPs which are insulating or semiconducting to achieve high conductivity range through subgap states that span between 10^{-12} to 10^5 S cm^{-1} . The control over both dopant (small spherical anions to larger polyions) type and concentration has been shown to modify the conductivity over several orders of magnitude by influencing their tertiary structure and carrier concentration (Singh and Singh, 2017; Chowdhury et al., 2018).

With the production of the highly conducting polyacetylene film towards the end of nineteen seventy; conductive polymers with π -conjugated systems have been vigorously investigated for synthesis of the next generation electronics, specifically organic electronics (Shirakawa et al., 1977). Presently, wonderful advances have been made in the molecular design and synthesis of an amazing variety of π -conjugated molecules. There is now strong tunability of the band structure and synthetic control, redox optical and electrochemical properties of organic semiconductors, at the molecular level (Pron et al., 2010). Organic electronics devices are being manufactured with the aim of cheap fabrication and enabling completely new device functionalities (like impact resistance, mechanical flexibility, and optical transparency that are difficult to achieve with their inorganic counterpart (Lin et al., 2012).

Deoxynucleic acid (DNA) is a biopolymer that comprises monomeric units called nucleotides. The nucleotides consist of three basic components; a nitrogenous heterocyclic base, a pentose sugar and a phosphate residue. There are many features both chemical and structural that make DNA an important scaffold for self-assembly of various 1-dimensional nanostructures. DNA can be found in various lengths ranging from nanometres to micrometres and with a high surface to volume ratio with a diameter of about 2 nm. This makes it suitable for controlled material growth since the length can be adjusted by increasing or decreasing the number of base pairs. It has many bonding sites at which materials can adhere. The ability of metal ions to interact and stick to DNA molecules has given rise to the synthesis of different kinds of 1-dimensional semiconducting nanostructures such as nanowires, nanopores and nanotubes. These involve the treatment of the DNA molecules with the appropriate metal ion leading to the doping of the DNA strand with the metal and subsequently reaction with the appropriate source of chalcogenide (anion) to precipitate on the metal leading to the formation of continuous coated 1-D nanostructures. This process makes use of the stiffness and metal-binding properties of DNA and is not dependent on the sequence information on the DNA (Anindya, 2020; Yang et al., 2018; Grumezescu, 2016).

Conductive polymers are attractive materials to template on DNA because together they form smooth, conductive nanowires (NWs) which provide access to a wide range of chemical functionality for subsequent applications. Whilst inorganic NWs may be functionalised using methods originally developed for bulk substrates, the polymer NWs

can be functionalised simply by derivatisation of the monomer and the density of functional groups adjusted by copolymerisation (Gualandi et al., 2021; Bai and Shi, 2022). Conductive polymers exhibit good electronic, magnetic and optical properties of both metals and semiconductors while retaining the attractive mechanical properties and processing advantages of polymers which makes them a distinctive class.

One dimensional nanostructure has attracted intensive research owing to their different applications in fabrication of nanoscale devices and in mesoscopic physics. These 1D nanostructures are important because they have discrete molecular states, show density of state singularities and exhibit special phenomena such as spin - charge separation¹⁵.

Among the different types of 1D nanostructure, nanowires are the most interesting and widely studied. Nanowires are particularly interesting in the investigation of charge transport at the nanoscale and have been studied for their applications as transducers in gas sensors or as interconnects in nanoelectronic devices. They can be synthesised using different methods including solution-phase techniques which allow high quality and large scale production (Paradisi et al., 2021; Wang et al., 2021).

The predicted growth in international energy application and the release of pollutants has led to a serious focus on advanced surveillance technologies for environmental protection; restoration and remediation (ITRS, 2011). Chemical sensors have become an important component of the technology controlled society. The high sensitivity, short response time, biocompatibility, economical synthesis procedure, and easy charge transfer properties of nanomaterial modified polyindole open up an appropriate choice for researchers to utilise these materials as a sensor. However, the applications of polyindole and its nanocomposites in sensing and environmental remediations remain challenging. The response and recovery time of sensors based on conductive polymers have shown remarkable enhancement with great advances in nanoscience. However, the selectivity toward specific analytes is still a challenge, therefore, it is essential to wisely functionalise these CPs with suitable receptors.

Further experiments on evaluating conductive polymer nanowires as gas sensors may result in exploring more of their potential applications which have not yet been investigated. In our previous work the chemical, morphological and electrical characterisation of the PIn/DNA nanowires, that were synthesised by chemical oxidation of indole and its subsequent mixing with DNA to form the DNA templated polyindole composite, have shown the formation of a smooth hybrid material with good electrical conductivity. In this research work we report the development of a gas sensor based on polyindole DNA (PIn/DNA) nanowires for the detection of volatile organic compounds (VOCs) at room temperature. Current output is proportional to the analyte concentration. Therefore, the need for this research which will evaluate the potential of DNA templated polyindole nanowire for indoor sensing of volatile organic compounds.

Materials and Methods

PIn/DNA nanowires were chemically synthesised using iron (III) chloride as an oxidant; as explained in my previous work (Yahaya, 2021), by dropping 5 μL of freshly prepared indole solution (6.24 mM) to 20 μL of λ -DNA (500 ng μL^{-1}) in the presence of 5 μL of MgCl_2 (0.5 mM), then 5 μL of FeCl_3 (1mM) dropwise into the solution. The solution was thoroughly mixed and allowed to react for at least 1h at room temperature. The chemical and morphological characterisation of the PIn/DNA were all discussed and explained in the work.

Sensing measurements

Polyindole DNA-templated nanowire films gas sensing test experiments were carried out by depositing 5 μL of the nanowire solution between Platinum electrodes (by molecular combing method) that has thin copper wires connected to their two terminal sides and allowed to evaporate at room temperature. The dry and wet air flow was controlled by Brooks's digital mass flow metre at room temperature in a Laboratory fume cupboard as shown in Figure 1. The gas characteristics of the PIn/DNA nanowire were investigated by recording their electrical responses when exposed to different gas mixture (dry air and vapour of the VOCs) that were passed at different flow rate. Amperometric Detection (AD) (Chronoamperometry) Palmsens.com software (with T equilibrium = 20s, Edc = 5 volts, t interval = 1.0s and t run = variable time) was used to acquire the sensing data.

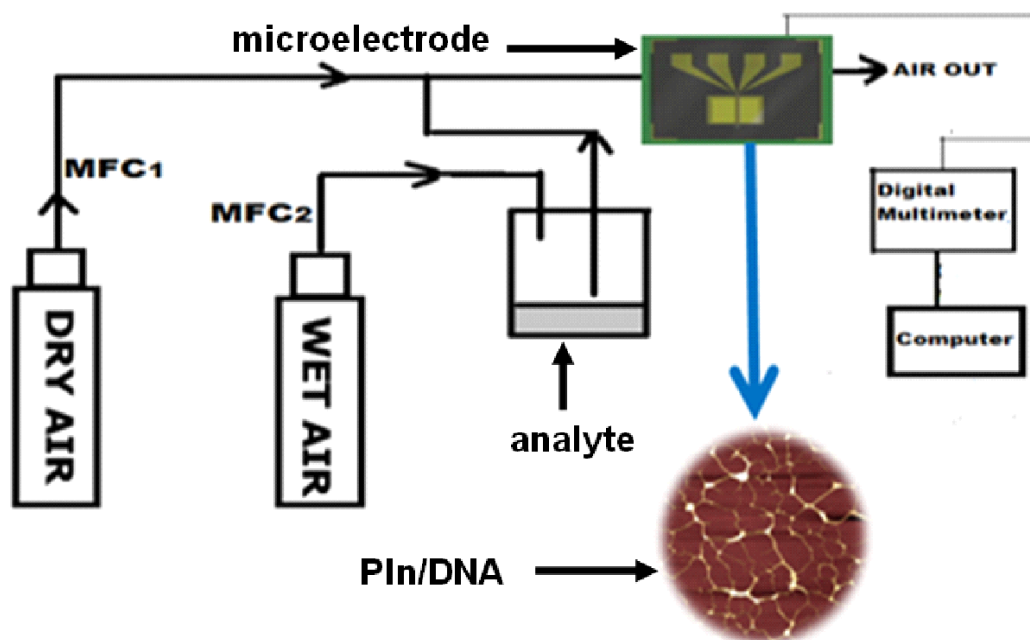


Figure 1. Schematic diagram of the laboratory fume cupboard gas sensing system.

The gas characteristics of PIn/DNA nanowire film were investigated by recording their electrical responses when exposed to different gas mixture (dry air and vapour VOCs) that were passed at different flow rate. The response of the sensor is defined as:

$$S = \frac{\Delta R}{R_0} \quad (1)$$

Where ΔR is the change in resistance of the device before exposure to the analyte for long enough that the resistance R has reached a steady-state and R_0 is the reference value of the sensor. The relationship between signal S and the way the analyte interacts with the sensor can be based on the reasonable assumption that only gas molecules which adsorb on the sensor can affect the resistance.

Results and Discussion

Data displayed in Figure 2a-d shows the response/signal output in relation to the concentration of the DNA-templated polymer nanomaterials, in all the charts there is an increase in current measured as the concentration of the bulk PIn/DNA nanowires increases. Mechanism of PIn/DNA sensing was proposed to be due to adsorption of the analyte and subsequent swelling of the polymer that change the value of polymer dielectric constant. This linear relationship between the current output and the analyte concentration may be due to the increase in number of analyte gas molecules that will be available for adsorption on the sensor which in turn increases the current (Lange et al., 2008; Li et al., 2007).

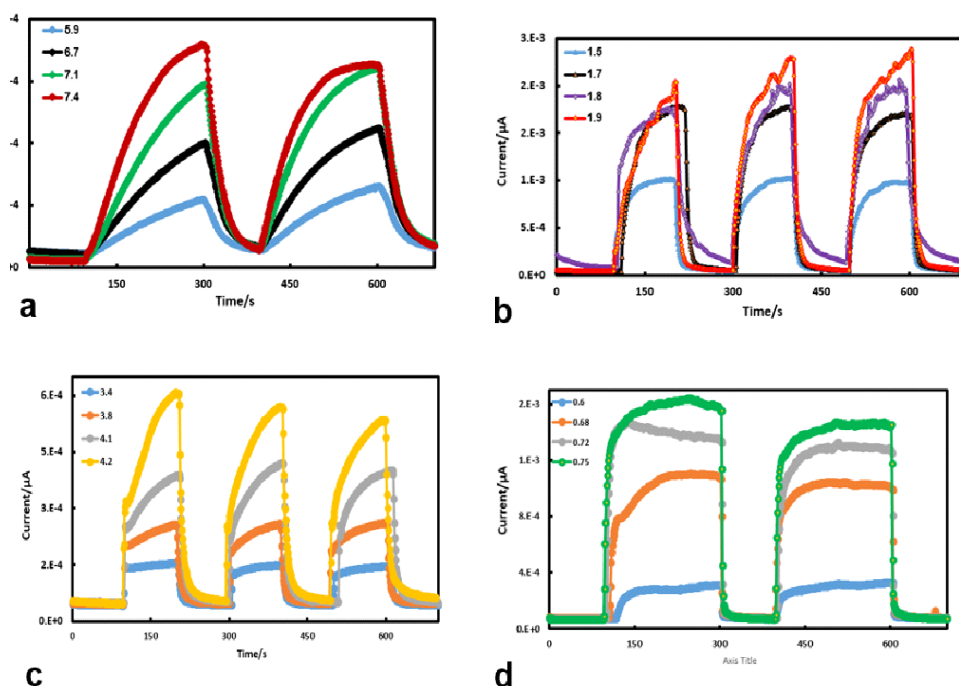


Figure 2. Current as a function of PIn/DNA concentration charts for different concentrations and cycles of VOCs sensing at 290K in (mmol/dm³): (a) two cycles of acetone sensing (b) three cycles of ethanol sensing (c) three cycles of Methanol sensing (d) two cycles of propanol sensing.

Repeatability and sensitivity

Figure 3 illustrates the general typical current transients of PIn/DNA nanowire device,

sensing sensitivity and repeatability at a constant biased voltage (the amount of voltage that an electronic device needs in order to power on and function) of 5 V. In due course of time, the current is switched forth in the presence of VOCs vapour and switched back to base current when the vapour is switched off, that is, only in the presence of air.

The response time is defined as the time it takes for conductance/resistance of the gas sensor to increase to 90% of the maximum conductance/resistance when a specific amount of gas was introduced into the sensor test chamber (written as t_{r90}). The recovery time (t_{d90}) is the time required for a 90% reduction in conductance/resistance when the gas is turned off and air was reintroduced into the chamber (that is, the time to reach 10% conductance/resistance in volatile organic compounds vapour) (Mouapi et al., 2021).

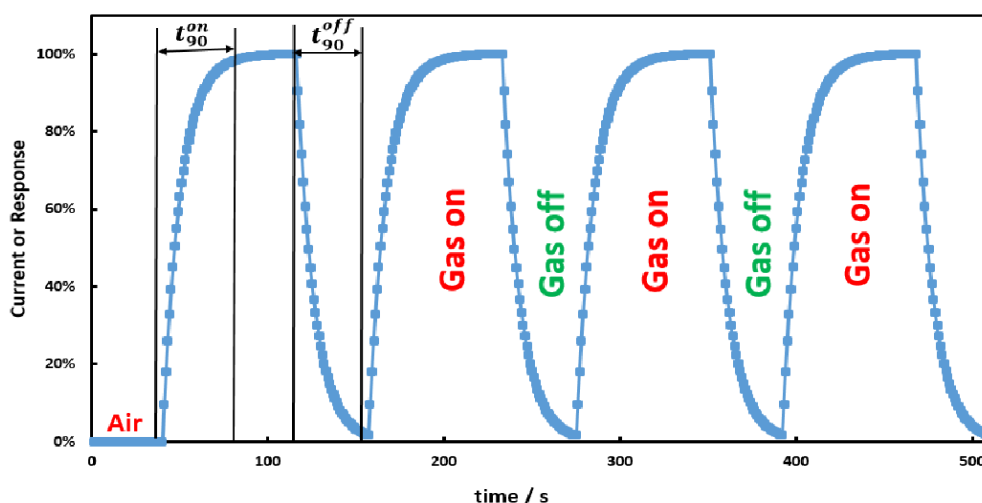


Figure 3. Representation graph of response-recovery time, sensitivity and repeatability of PIn/DNA templated nanowires-array based gas sensor towards different volatile organic compounds at different Kelvin temperatures.

Figure 4 displayed the response time for the PIn/DNA nanomaterials towards the VOCs analytes tested; from the chart chloroform (104s) takes the longest time to respond when exposed to the PIn/DNA bulk nanowires, followed by methanol with a response time of 103s. Acetone and propanol respond to the polymer composite films at the same rate (99s) while ethanol and hexane both have responses time of less than 80s.

From the same Figure 4, ethanol (65s) was recovered faster than all the other VOCs while acetone (93), chloroform (98) and methanol (91) took more than one and half minutes to be recovered. For propanol and hexane their recovery times are less than one and a half minutes. Overall, response time is very fast in comparison with the recovery time. The expeditious and straight forward response makes the PIn/DNA nanostructures suitable for practical applications in VOCs sensing. According to different researchers, the performance of 1-D nanostructured sensors depends greatly on the chemical composition, microstructure, processing techniques, crystallinity, surface morphology, sensor fabrication arrangements, operating temperature and humidity (Neri, 2017; Li et al., 2017).

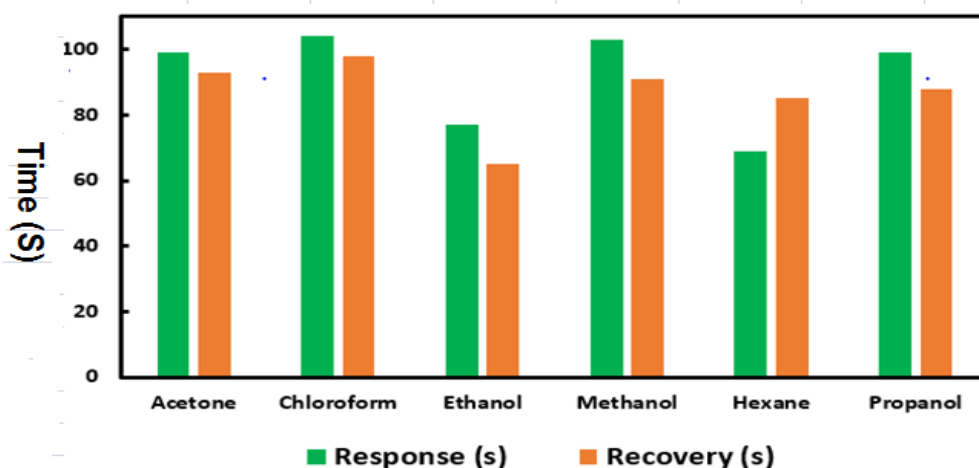


Figure 4. Response (t_{r90}) and recovery (t_{d90}) times for PIn/DNA nanomaterials.

To check the repeatability of the PIn/DNA nanowires sensing test for the VOCs, their response ratio (the change in current in the presence of the analyte vapour) was calculated and presented in Figure 5. The response ratio was calculated using the formula below (equation 2):

$$\text{Response ratio} = \frac{\Delta I_n}{\Delta I_1} \quad (2)$$

Where, I_n is the change in current of the last cycle response for the sensing test for an analyte and I_1 is the initial change in current for the first cycle response. A ratio value of close to 1 indicates a good response or repeatability, which is a desirable sensor characteristic. The response and baseline ratios were calculated for four ($n = 4$) sensing cycles for all the analytes and displayed in Figure 5.

From the chart for response time ethanol has the highest ratio (1.0) followed in descending order by propanol (0.96), chloroform (0.93) and acetone (0.92). Methanol (0.73) and hexane (0.71) have the least values among the VOCs sensed. Generally, all the analytes have a response ratio in the range of 1 – 0.71, which is an indication of some reversibility/repeatability.

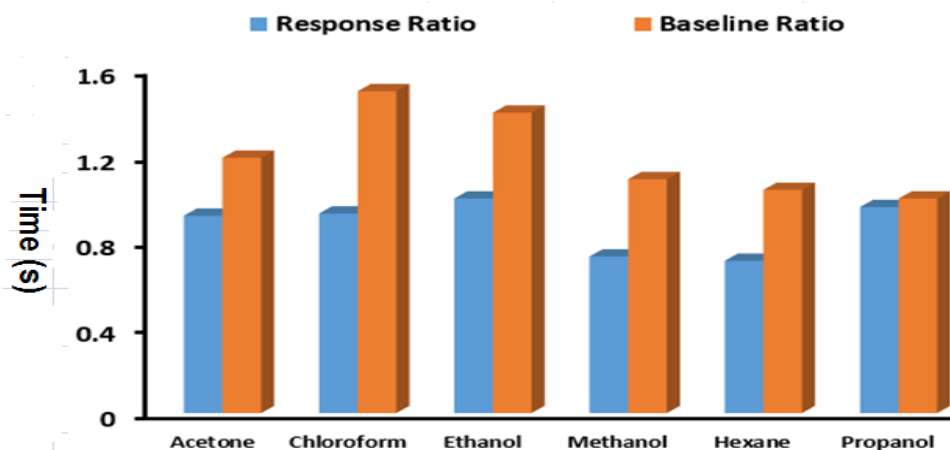


Figure 5. Response and baseline ratios chart for volatile organic compounds sensing in the PIn/DNA nanowire.

The baseline consistency during the sensing test at different cycles for the analytes vapour were analysed using the background ratio formula in equation (3):

$$\text{Background ratio} = \frac{B_n}{B_1} \quad (3)$$

Where, B_n is the baseline value in the last response/recovery cycle for the sensing test for an analyte and B_1 is the initial baseline value.

The highest baseline drift value was observed in chloroform (1.5) while the least of 1.0 was recorded for propanol. Generally, the baseline is stable for all the analytes detected except for chloroform and ethanol that show slightly higher values and a drifting baseline. The observed results above may be due to the chemistry of the analytes.

Sensitivity is the ratio of the incremental change in the sensor's output (Δy) to the incremental change of the measure input (Δx). The slope of the calibration curve, $y = f(x)$, can be used for calculation of sensitivity (Peterson et al., 2017; Stefan et al., 2017).

Figure 6 display the slope of the calibration curve for PIn/DNA nanowires towards different VOCs sensed in this work. The responses most likely are due to adsorption of the molecules on the composite and then changes in the polymer conformation.

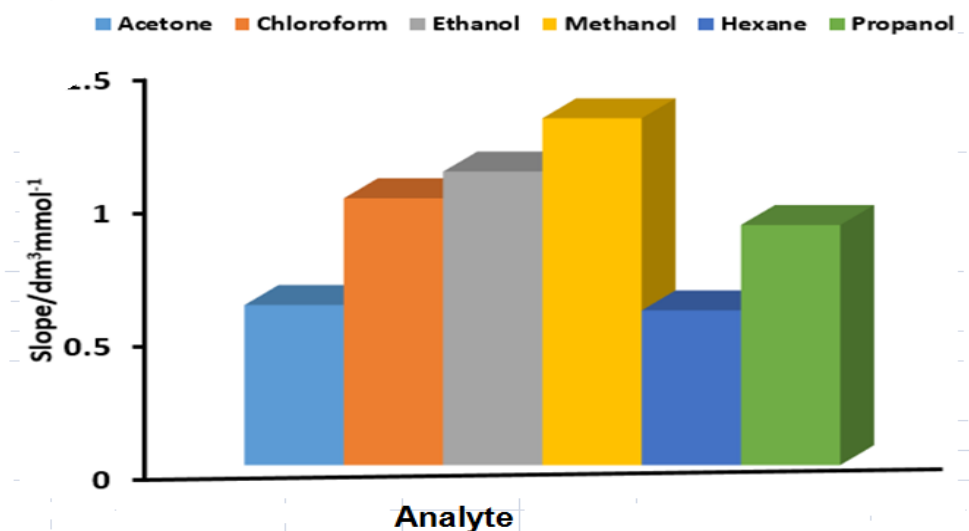
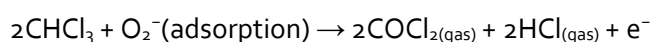


Figure 6. Slope of the calibration curves (sensitivity) for the different gas analytes illustrating the sensing behaviour of the prepared PIn/DNA nanowires samples under different VOCs.

From the slope of the calibration curves in Figure 6 all the analytes have sensitivity close to unity with the exception of methanol, ethanol and chloroform that have values greater than or equal to one. Previous experimental results, demonstrated that some analyte gases, especially alcohols and ketones, can change the crystallinity of conducting polymers (Šetka et al., 2017; Nag et al., 2017).

Hexane (0.58) has the lowest sensitivity towards the PIn/DNA nanowires, which may be said to be due to its low/weak adsorption of the analyte at room temperature and under warm conditions. The low detection may be also because of the weak physical interactions between it and the sensing PIn/DNA nanowires, involving its adsorption or swelling. Even though these interactions do not change the oxidation levels of the conducting polymers, it can influence the properties of the sensing materials (Park et al., 2017).

If the sensor is exposed to a reducing gas such as chloroform the reaction between the vapour and the oxygen species result in a decrease in the O_2^- or O^- ion concentration on the surface and consequent increase in the concentration of the electrons. This decreases the resistance of the polymers. Therefore, the adsorbed species play an important part in sensing chloroform as given by the following reactions:



Generally, the major sensing features aimed for in vapour/gas sensitive materials include sensitivity, selectivity, stability, and speed of response; features referred to as the four "S"s of gas sensing. Furthermore, these materials are expected to interact reversibly and reproducibly with the specific analyte of interest showing strong stability in different environmental conditions. From the results obtained in this research, PIn/DNA composite have displayed good sensitivity (close to unity), selectivity, stability, and speed of response (greater than 60%) to most of the VOCs tested therefore, it can be considered as suitable materials for the development of VOCs room temperature nanosensor.

Conductive polymers sensing mechanism

In chemical sensors concentrations of the analytes were transformed to other detectable physical signals which were due to interaction between the sensing element and the analyte. The mechanism of conductive polymers nanowires sensing can be explained in terms of the interactions between gas molecules and conducting polymer films. The physical properties of conducting polymers strongly depend on their doping levels, which can be changed by chemical reactions with many analytes. Resistance and work function of sensing material can be affected by electron transferring. When detecting electron donating gas an opposite process will occur (Bai and Shi, 2007; Ram et al., 2005).

Except for the pure redox reactions, partial charge transferring also leads to an alternation in the conductivity of a conducting polymer. Direction of partial electron transfer can be determined by the electronegativity of the analyte and the work function of the polymer while in some it occurs in the polymer mixture. Swelling and dissolving of counter ions of the polymers into the analyte are other popular processes that were used by different researchers to explain conductive polymers sensing mechanisms (Dixit, 2005; Athawale et al., 2006).

Generally, for p-type sensing material, a remarkable amount of oxygen molecules from the air are initially chemisorbed on the surface, causing electrons to be removed from the conduction band. The chemisorbed oxygen molecules are then transformed into a single or double oxygen ion, becoming absorbed on the surface. The electron density decreases due to the removal of electrons while the hole concentration increases. Thus, this causes a decrease in resistance.

When reducing gas such as ammonia reacts with the adsorbed species, electrons are donated to the conduction band of the p-type material, after while reducing the whole concentration due to electron hole combination. As a result, the resistance increases. For oxidising gas such as nitrogen dioxide, electrons are withdrawn and depleted from the valence band which results in increase in hole concentration and resistance decreases. The sensing mechanism becomes conversely for the n-type material. Holes and electrons are responsible for conduction in p-type and n-type semiconductors, respectively (Tang et al., 2018).

Figure 7 shows the conventional gas sensing mechanism of p-type gas sensors in response to air and ammonia, respectively. In explaining the gas sensing mechanism of the conducting polymer sensors doping and undoping play vital roles. 'P-type doping (that is doping with oxidising agent) introduces charge carriers into the electronic structure and forms p-type conducting polymers. On the other hand, the use of a reducing agent corresponds to n-type doping (Patil et al., 2019).

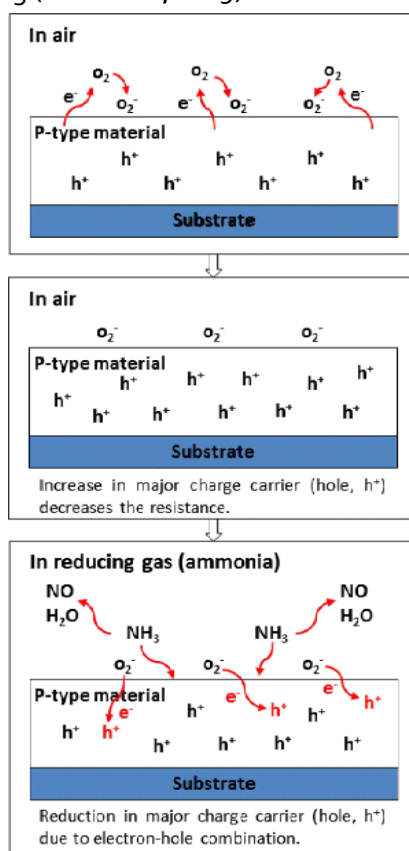
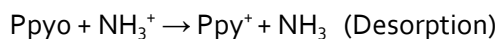
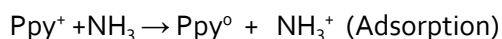


Figure 7. Mechanism of p-type material sensing in air and reducing gas medium.

Source: Wong et al. (2019).

Specifically, the sensing response to various analytes is affected by the oxidation level of the conducting polymer, which is likewise readily affected by either chemically or electrochemically doping/undoping mechanisms between the interacting analytes and the conducting polymer. Protonation or redox reactions are responsible for the doping and dedoping of most conducting polymers. The doping level is alterable by transporting electrons to or from the target analytes. Changes can be induced in work function and resistance of the sensing materials by electron transport. The work function refers to the minimum energy required to remove an electron from bulk to vacuum level (Wang et al., 2021).

For instance, when polypyrrole (Ppy) reacts with electron-donating gases such as ammonia, the whole concentration reduces and the resistance increases. On the flip side, analytes such as alcohols serve as electron acceptors that form new holes within the Ppy structure, leading to a decreased electrical resistance (Zhang et al., 2021). Nevertheless, purging the sensing layer of Ppy with dry nitrogen or air can partially or fully recover its resistance. The possible reactions involved in ammonia sensing are as follows:



The mechanism above showed that the p-type polypyrrole is expected to be undoped under an ammonia environment. During ammonia sensing, a single pair of nitrogen electrons donated to the initially oxidised conductive polymer causes a decline in electrical conductivity, forming a neutralised polymer one possible reversible interaction mechanism between the polymer (Ppy) and the electron donor (ammonia) is the decrease in doping level of the Ppy through the compensation of the original dopant effect. The desorption of gas molecules involves a proton transfer between the Ppy and NH_3 , where the ammonia can easily attack the protons of Ppy, forming ammonium ions. In summary, the doping process is crucial in enhancing conductivity of the polymers, in which the conductivity will affect the gas sensing mechanism and performance (Centane and Nyokong, 2021). For this study it was believed that the mechanism of Pln/DNA sensing is by absorption of the analyte molecules on the surface of sensing film.

Conclusions

For the response time, chloroform (104 Secs) takes the longest time to respond when exposed to the Pln/DNA bulk nanowires while ethanol and hexane both have responses time of less than 80 Secs. While for the recovery time, ethanol (65 Secs) was recovered faster than all the other VOCs while acetone (93 Secs), chloroform (98 Secs) and methanol (91 Secs). Overall, response time is very fast in comparison with the recovery time. Measurement of response time revealed ethanol to have the highest ratio (1.0) and hexane

(0.71) have the least values. On the other hand, the highest baseline drift value was observed in chloroform (1.5) while the least of 1.0 was recorded for propanol. Finally, for the sensitivity Hexane (0.58) has the lowest towards the PIn/DNA nanowires, while methanol has the highest (1.3). Conclusively, it can be said that the PIn/DNA nanowires have the potential to be used as sensor materials for VOCs at room temperature as all their responses to the different sensor parameters were in compliance with that of a good working sensor.

Conflicts of Interest

The author declares that there is no conflict of interest regarding the publication of this paper.

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