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CONJUGATED POLYMER/MULTI-WALL CARBON NANOTUBES COMPOSITE BASED VOLATILE ORGANIC COMPOUNDS SENSORS

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ABSTRACT

Polyimidazole multi-walled carbon nanotubes (Plm/MWCNTs) nanocomposite films were synthesised by chemical oxidation of imidazole monomer and addition of MWCNTs to the conductive polymer (CP) solution in a 1:1 ratio. The sensing performance of the Plm/MWCNTs nanocomposite films was measured by recording their electrical response when exposed to contrasting mixtures of gas. Generally, the nanocomposite film with a diameter of 3-12 nm shows a fluctuating response of their resistance when exposed to the analyte together with an excellent sensitivity of 90%. The response is quantified as the fractional change in resistance compared to the baseline, R_0, i.e., the response ratio $S = \Delta R/R_0$. Values of S in the range 0.60–1.5 were observed for volatile organic compounds. This research work provides a simple, economical and environmentally-friendly technique to create favourable Plm/MWCNTs composite based volatile organic compounds sensors, which could be employed for environmental monitoring in workable electronic devices in domestic and industrial applications. The research findings established that Plm/MWCNTs nanocomposites have promising future applications in gas sensing.

Key words: Sensors, Composites, Polymer, Carbon Nanotubes, Polyimidazole

INTRODUCTION

The development of effective chemical detectors with high perceptivity, trustability, less cost and bitsy size needs abecedarian knowledge of chemistry associated with advanced detection approaches. The electrical resistance change during a sensitive membrane in the presence of chemical species is covered in chemiresistive detectors, which is associated with the analyte consolidation.

Chemiresistors have drawn a lot of interest due to their ease of assembly and lucidity (Ryabchikova, 2021; Korotcenkov, 2020).

They have been used to detect and monitor volatile organic compounds (VOCs) in numerous environmental monitoring, biomedical applications, vehicle and industrial emission control, and household security (Abdulsamad, 2021).

For decades, one-dimensional nanostructured accoutrements have gained growing interest and wide use in different experimentation fields like electronics, detectors, optics, information storehouse, biomedical, catalysis, and energy conversion. Carbon Nanotubes (CNTs) are long tubeshaped carbon nanomaterials made of only carbon with a wideness of a couple of nanometers. They can be classified as single-walled (SWCNT) or multi-walled (MWCNT) that contain several concentrically inter-linked nanotubes. Carbon nanotubes have established wide use in numerous fields. They can be mixed or blended into other matrix substances like polymers and form nanocomposite structures of high strength, low weight, and straightforward face functionalisation (Wang and Gu, 2021).

In the sensing application, carbon nanotubes appeared to have a weak response to sense chemical vapour, for example, acetone and propanol. It has been informed that the sensitivity of carbon nanotubes increased after decorating with nanoparticles such as Ag or by adding different types of polymer such as polypyrrole. Mixes of conjugated polymers are getting more application in organic electronics tools due to the electronic and optical behaviour of the polymers, which are similar to those of semiconductors (Sreenath *et al.,* 2020; Kausar, 2016).

A nanosensor is a device capable of carrying data and knowledge about the properties and characteristics of molecules at the nanoscale

position (Rani and Ray, 2021). They are used to determine chemical or mechanical information like the presence of chemical species and nanoparticles or cover physical parameters like temperature. They are specific or selective in their ability to detect a number of analytes. Some nanosensors may have the power to detect one sort of analyte (called singlex). Some can detect many analytes and are known as multiplexes (Holthoff and Stratis-Cullum, 2010). The chemical sensors supported by nanocomposites of conducting polymers with carbon nanotubes have gained considerable interest for having better detection performances. The physical and chemical characteristics of conducting polymers can be explosively changed by the addition of CNT because of the colourful interfacial relations between them. Lower discovery limits, increased superficial area, low resistivity or faster response times, and improved environmental stability are among the benefits of nanocomposites of conducting polymers with CNT once they're used as sensor elements. These functions show the selectivity and specificity of nanosensors due to their recognition of elements (Bhandari *et al.,* 2021).

The manufacture of pharmaceuticals, paints and refrigerants are among the significant artificial sources of many VOCs. They are among the major contributors to air pollution. The monitoring of these pollutants for human health, as they can cause several diseases, is an important aspect of research, and in fact, the detection of toxic species is an issue of growing interest (Pitiriciu and Tansel, 2021).

Evaluating the ecological effect of natural pollutants needs systematic tools which will expeditiously screen them with a minimal sample. Therefore, the look for sensitive, highly selective, stable, robust and low-cost sensors for VOCs is a field that has received tremendous interest from many researchers since they are expected to perform a crucial role in environmental assessment. Thin-film sensors at low gas concentration have the disadvantages of low response and recovery, complex fabrication, low repeatability, high-temperature operation, poor sensitivity, large sample size and non-tunable charge transport properties as indicated in their electrical behaviour. Notwithstanding, gas sensor research is still developing and has yet to reach its full prospect in competence and operation. Thus, there are powerful demands for highly selective, sensitive, cost-effective, stable, and vigorous VOC nanosensors (Lee *et al.,* 2020; Kolosnjaj-Tabi and Moussa, 2017). Therefore there is a need for this research work.

In this research, imidazole was used to coat MWCNTs to study the sensing properties of CNTs at room temperature. The films were prepared by drop-casting. Imidazole is an organic compound with a 5-member ring. The chemical formula is $C_3H_4N_2$ and can be classified in the aromatic heterocycle group. It has been reported that imidazole can be found in natural compounds such as alkaloids; because of its unique structure, imidazole is a promising material in pharmaceutical applications. In addition, it has many industrial applications, for instance, as an anti-corrosion special with Cu because Cu missed its conductivity due to corrosion (Agarwal, 2021).

EXPERIMENTAL

Materials

Imidazole ($C_3H_4N_2 \geq 99$) was bought from Sigma-Aldrich Company Ltd.; Ferrous sulphate heptahydrate ($FeSO_4$. $7H_2O$, 99%), CNTs were provided from Thomas Swan Company, (Elicarb™, England) and Magnesium Chloride ($MgCl_2$. $7H_2O$, 99%) were purchased from BDH. Lambda DNA was bought from Biolab UK. All the chemicals were of Analar grade.

Normal Silicon wafers were bought at Compart Technology UK. Microelectrodes were supplied by Windsor Scientific Ltd. The bandwidth was 10 μm, the inter-electrode gap was 10 μm, and the electrode length was 2 μm. Each Pt-MB4000 device comprises 4 independent microbands and 4 independent contact pads. Copper grids were bought at EM Resolutions UK for TEM analysis. Deionised water was used for all the experiments.

Substrates cleaning

Using a diamond scribe, wafers n-Si were divided into ~1 x 1 cm^2 portions and submerged in water-diluted acetone for about 50 minutes. Then rinsed with distilled water, dried in Nitrogen gas, and further dried in an oven at 35 ^{o}C for 5–10 min.

FEMTO plasma system (Diener electronic, Germany) was used for final cleaning, using oxygen gas oxidation (15 min, 90 W, 15 sccm) at 40% power.

Chemical synthesis of Polyimidazole

Polyimidazole was synthesised using $FeCl_3$ as the oxidant. Typically, 5 µL of newly prepared $C_3H_4N_2$ (solution (1 mM) was added to 5 µL of $MgCl_2$ (0.5 mM), then 5 µL of $FeCl_3$ (1 mM) was added dropwise into the solution. The mixture was vigorously mixed and allowed to react for 2 h at room temperature, as explained by Al-Hinai *et al.* (2016). The reaction was accomplished by the disappearance of the white coloured imidazole in the mixture to a colourless homogeneous mixture.

Preparation of CNT/Plm Composite

MWCNTs was mixed with 10 mL of methanol and sonicated (2 - 3) h to disperse the nanotubes. 20 µL of the prepared polyimidazole was added to 20 µL of MWCNTs (1:1), and the solution was left for 2 h. The Plm/MWCNTs mixture was used for the sensing tests.

Sensing process

Polyimidazolemulti-walled carbon nanotubes (Plm/MWCNTs) nanocompositefilm gas sensing test experiments were carried out by depositing 5 µL of Plm/MWCNTs solution on a platinum electrode (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 airflow was controlled by Brooks's digital mass flow metre at room temperature in a Laboratory fume cupboard. The gas response/or sensitivity of Plm/MWCNTs film were probed by recording their electrical responses when exposed to the different gas mixture (dry air and VOCs vapour) that were passed at a different flow rate. Amperometric Detection (AD) (Chrono-amperometry) with a Palmsens.com software (where T equilibrium = 20 s, Edc = 5 volts, t interval = 1 s and t run = variable time) was used to acquire the sensing data.

The sensitivity was calculated from equation (1):

$$S\% = \frac{(I - I_o)}{I_o} * 100\% \qquad (1)$$

Where S% is the sensitivity of the film, I_0 is a current before vapour exposing and I the electrical current after gas exposure.

The sensor response is defined as:

$$S = \frac{\Delta R}{R_0} \qquad (2)$$

where ΔR (R-R_0) is the difference in resistance of the device before subjection to the analyte for long enough that the resistance R has reached a steady-state and Ro is the reference value (baseline or resistance of the device before exposure to the analyte) of the sensor. In the case where a steady-state resistance is not reached, it is usual to choose the value for ΔR to be equal to that after a particular time, e.g. at the 30 s or 60 s of exposure.

RESULTS AND DISCUSSION

Sensing results

In an irreversible sensor, the base current recovers partly, while in a flexible sensor, it is fully recovered. Reversibility is crucial for applying a gas sensor because most applications (urban pollution, industrial safety) require continuous monitoring of gas levels rather than a single-use device as in, for example, blood glucose analysis (Hamid and Çelik-Butler, 2018; Yamamoto and Uno, 2018).

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. A further assumption, which is reasonable, but not necessarily accurate, is that the signal is proportional to the amount adsorbed per unit area (Naeimi *et al.,* 2019).

Figure 1 shows the general representative current transients of Plm/MWCNTs film device, sensing sensitivity and repeatability at continuous biassed voltage. The reproducibility in the response can be ascribed to the adsorption and desorption of the analyte vapours during exposure and removal of vapours.

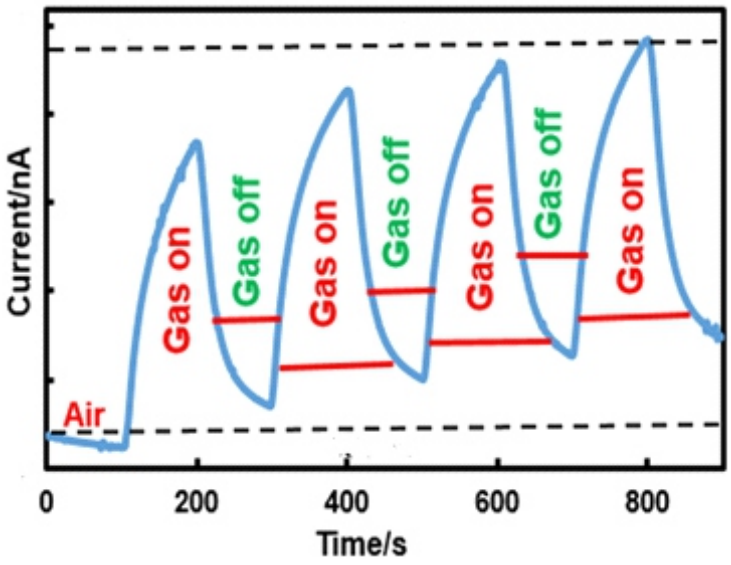

Figure 1: Schematic graph of sensitivity and repeatability of Plm/MWCNTs composite gas sensor towards different volatile organic compounds at a given Kelvin temperature.

From Figure 2, the major framework for gas sensors, the response and recovery times was determined. The response time is the time it takes for the resistance of the sensor to extend to 90% of the utmost resistance when a given quantity of gas is introduced into the detector test chamber (written as t_{r90}). The recovery time (t_{d90}) is the time needed for a 90% reduction in resistance when the gas is turned off and air is greeted into the chamber; that's the time to reach 10% resistance inorganic compounds vapour (He *et al.,* 2019).

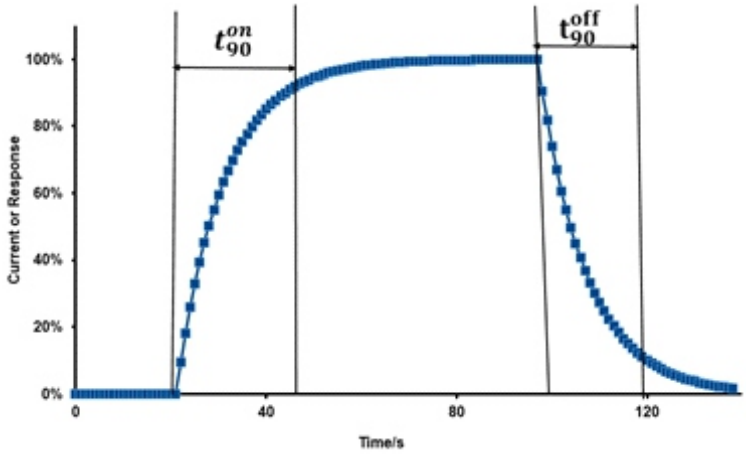

Figure 2: Schematic graph of response-recovery time

From Table 1 below, propanol has the fastest response and recovery (15 and 7 seconds, respectively) towards Plm/MWCNTs film sensors. The fastest responses towards the sensor are observed in propanol (15 s), methanol (16 s) and acetone (19 s), while the least responses are from chloroform, dichloromethane and ethyl acetate with 56, 54 and 52 seconds, respectively.

Table 1: Response and Recovery times and selectivity for Plm/MWCNTs based VOCs based Sensor.

Analyte	Concentration (Kpa)	Temperature (K)	Response Time (s)	Recovery Time (s)	Selectivity (%)
Acetone	0.67	RT	19	09	0.13
Chloroform	"	"	56	18	0.09
Dichloromethane	"	"	54	16	0.07
Diethylether	"	"	24	22	0.07
Ethanol	"	"	30	20	0.18
Ethyl Acetate	"	"	52	10	0.40
Methanol	"	"	16	20	0.18
Petroleum Ether	"	"	19	46	0.04
Propanol	"	"	15	07	0.12
P-Xylene	"	"	44	24	0.03
Toluene	"	"	20	06	0.06

Generally, the recovery is faster than the response of the VOCs towards the Plm/MWCNTs sensor. For the recovery time, the fast VOCs to be recovered in the sensor were propanol (7 s), acetone (9 s) and ethyl acetate (10 s). On the other hand, petroleum ether, P-xylene and diethyl ether with 46, 24 and 22 seconds, respectively, were the least recovered.

The particular reaction of the sensor towards a particular mark gas in a group of gases or the particularity of the gas sensor towards a particular target gas molecule in a mixture of gases is determined by its selectivity. From Table 1, the Plm/MWCNTs gas sensor has the best selectivity for ethanol and methanol gases (0.18%), while its selectivity towards *p*-xylene, toluene, dichloromethane and diethyl ether is very low (between 0.03 to 0.07%).

Response and recovery times of gas sensing film are determined by the thickness of the film which is a well-known fact that is closely associated with the distance of the diffusion path of the analytes. The possible reason for the recovery and response action is that at a better speed the film becomes thinner which causes a shorter diffusion length of analyte molecules and thus results in shorter recovery and response times (D'Arsié *et al.,* 2018).

Sensitivity and Concentration

Bare MWCNTs and Plm/MWCNTs sensitivities and effects of concentration were compared, and the results are displayed in Figures 3 and 4, respectively. Generally, the sensitivity raised with concentration increases in both substances except for petroleum ether on Plm/MWCNTs composite sensing that gives the reverse case, which may be due to its chemical structure. Sensitivities of VOCs are higher in Plm/MWCNTs composite than in bare MWCNTs. Ethanol (1.64) and methanol (2.50) have the highest sensitivity in bare MWCNTs and Plm/MWCNTs, respectively. On the other hand, *p*-xylene has the lowest sensitivity in both bare CNTs and Plm/MWCNTs. The observed behaviour can be attributed to the increased number of conducting molecules that increase electrical resistance, as Khalid *et al.* (2021) explained.

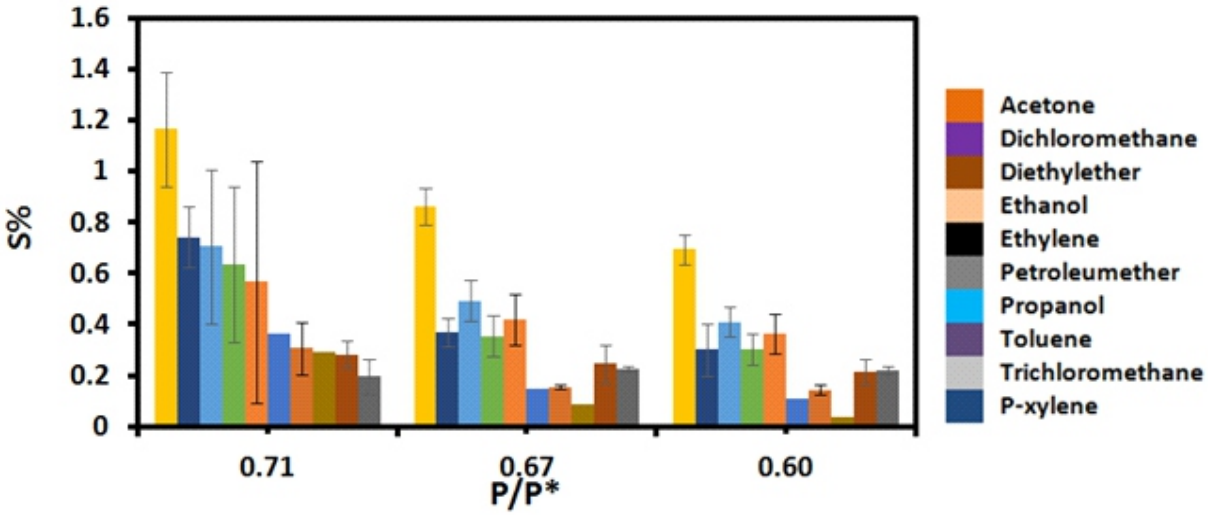

Figure 3: Bare MWCNTs sensing of VOCs

Resistance of nanocomposites materials rises with conductive fillers when exposed to gases which may be ascribed to the demolition of the conductive filler network due to the polymer inflammation (Pandis *et al.,* 2011). So many conductive polymers utilise assimilation/de-assimilation by redox and swelling at room temperature (Chiou *et al.,* 2019).

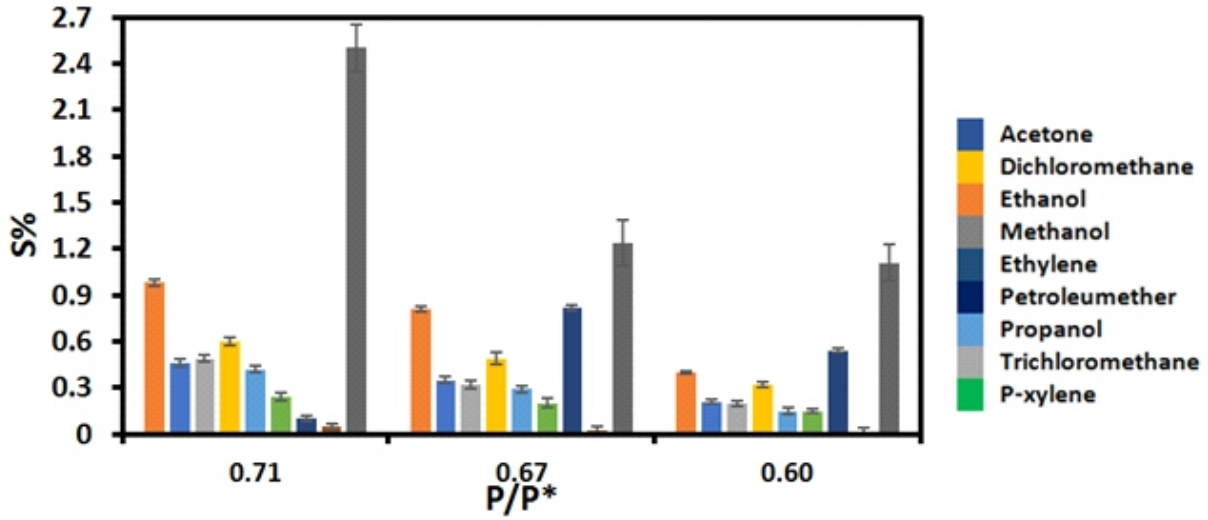

Figure 4: Plm/MWCNTs composite sensing of VOCs

When mixed with the target analyte and its sorption molecules, the polymer nanocomposite film will result in the swelling of the polymer matrix within the sensing medium, which supports the inflammation effect. This causes an increase in the volume of the polymer resulting to an increased distance between the adjacent conductive nanofillers depressing the conductive flow network formed by the filler within the polymer matrix, which, therefore, results in the rise in electric resistance. It's crucial to pick the proper nanofiller-polymer pair and ratio also because of the production procedure for effective use of the sensing mechanism (Tanabi and Erdal, 2019).

In spite of the significance of the sensing procedure, there's a robust basic desire to know the role of inflammation on the mechanical and electrical characteristics of polymer-based composites due to their sophisticated microstructures. Other sensing mechanisms are doping–dedoping, electron transfer and protonation–deprotonation mechanisms (Ehsani

et al., 2021).

Chiou *et al.* (2019) found that the operating temperature and acetone vapour concentration greatly affected the response of their polyethylene glycol multi-wall carbon nanotubes mixture sensing material. They suggested that the acquisition of sensor resistance may be an unambiguous procedure that gives information about the kinetics of atmospheric oxygen adsorption, electron-withdrawing or donating molecules through Van der Waals force, and acceptor-donor interactions. They believed that the great interaction of the polyethylene glycol surface with the target gas molecules initiated the charge transfer in multi-wall carbon nanotubes because the acetone response was increased as a function of the concentration of polyethylene glycol. Their sensor response to acetone was reduced at a short temperature, which was attributed to the weakly dipolar and chemical bond properties of the physically absorbed acetone on polymers.

Mechanism of Carbon Nanotube Composite Sensing

In chemical sensors, concentrations of the analytes were transformed into other detectable physical signals due to interaction between the sensing element and the analyte. The mechanism of conductive polymers/carbon nanotubes nanowires sensing can be explained in terms of the reaction between gas molecules and polymer films (Yuan *et al.,* 2020).

A delicate layer is deposited on the surface of a Platinum electrode to manufacture a chemiresistive sensor. The efficiency of the sensor is measured by observing the changes in electric resistance of the complex layer in the presence of an analyte in the course of which the potential of the sensing layer switch because of the interaction with the substance, requiring a sign associated with the sample concentration (Yang *et al.*, 2019).

The agreement of the nanofillers and polymer is an issue that must be considered within the design of polymer nanocomposites to get a nanocomposite with the specified sensing properties. Size, type, and nature as well as electric strain behaviour that is closely associated with the quantity and distribution of fillers within the polymer, are the factors that determine the properties of polymer nanocomposites (Sliozberg *et al.*, 2021). The strain theory evaluates the consequences of internally connected nanofillers in nanocomposites on electric conductivity that reduce with decreasing filler volume fraction up to the critical value (Pang *et al.*, 2020). Additionally, to make new material with certain functionality, a fundamental understanding of physical and chemical properties, knowledge about different interactions, and the prediction of structural influence on composite behaviour are mandatory. Matrix stiffness which will have an immediate effect on sensing performance thanks to analyte induced swelling of the matrix, is a crucial parameter which must be considered for the composite due to the contact of polymers with sample fluid gives a volume expansion because of the sorption, which mainly rely on the polymer's solubility within the sample (NSF, 2022).

The sensing technique that supported the swelling effect is displayed in Figure 5, where the polymer admixture film is exposed to the target analyte, and the sorption of sample molecules causes the inflammation of the polymer matrix. Consequently, the polymer quantity increases, leading to an increased length between the adjacent conductive nanofillers destroying/disturbing the conductive oozing network formed by the filler within the polymer mould, which ends up within the increased electric resistance (Leng and Lau, 2011). It's important to pick the proper nanofiller-polymer pair and ratio also because of the preparation procedure for effective use of the sensing mechanism. In spite of the importance of the sensing technique, there's a robust fundamental need for understanding the influence of swelling on the electrical and mechanical properties of polymer-based admixture due to their complex microstructures. Doping–dedoping, protonation–deprotonation and electron transfer are several other sensing mechanisms (Xu *et al.*, 2020).

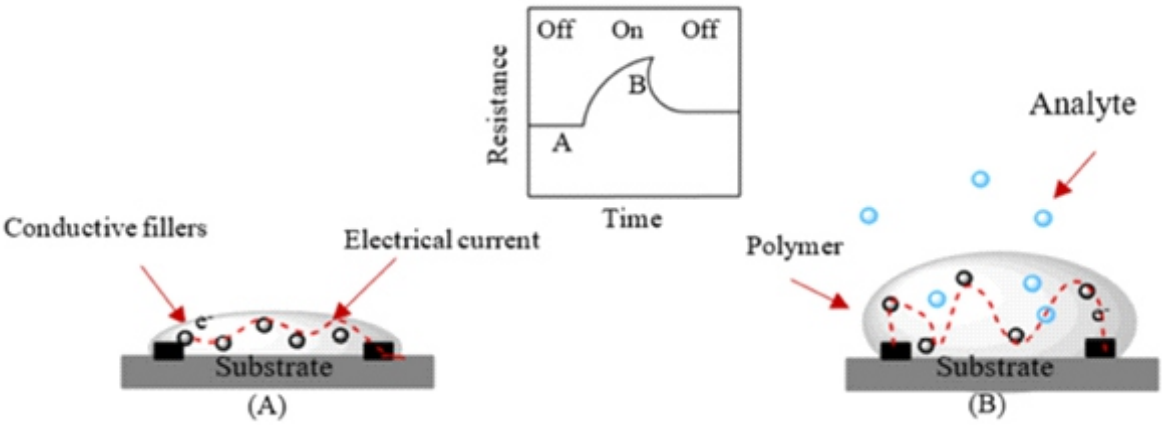

Figure 5: Diagramatic description of the chemiresistor procedure supported by the swelling effect. Red dashed lines indicate the electrical current along the flow pathway (A) before swelling (B) after swelling (Ehsani *et al.,* 2021).

Due to inter-nanotube contact resistance, the theory of charge conduction in composites using conductive polymers may differ. When CNTs are dispersed during a mould material, electrically connected systems of CNTs are formed, which can give three continuous conductive pathways, as shown in Figure 6. However, between the carbon nanotubes and at the contact points of the carbon nanotubes junction, a skinny non-conductive layer could also be formed.

Now, a carbon nanotubes network has two sources of electric potential, that is, the intrinsic resistance along the carbon nanotubes itself and, therefore, the contact potential at a nanotube junction because of a niche or cross-over between CNTs as shown in Figure 6. When the diameter of the inter-nanotube mould region is sufficiently small, the electrons can pass this region by the quantum mechanical tunnelling effect for conduction. A thick coating of polymer around carbon nanotubes leads to poor electrical conductivity, and conduction within the composite films is dominated by the tunnelling effect (Kuronuma *et al.,* 2012).

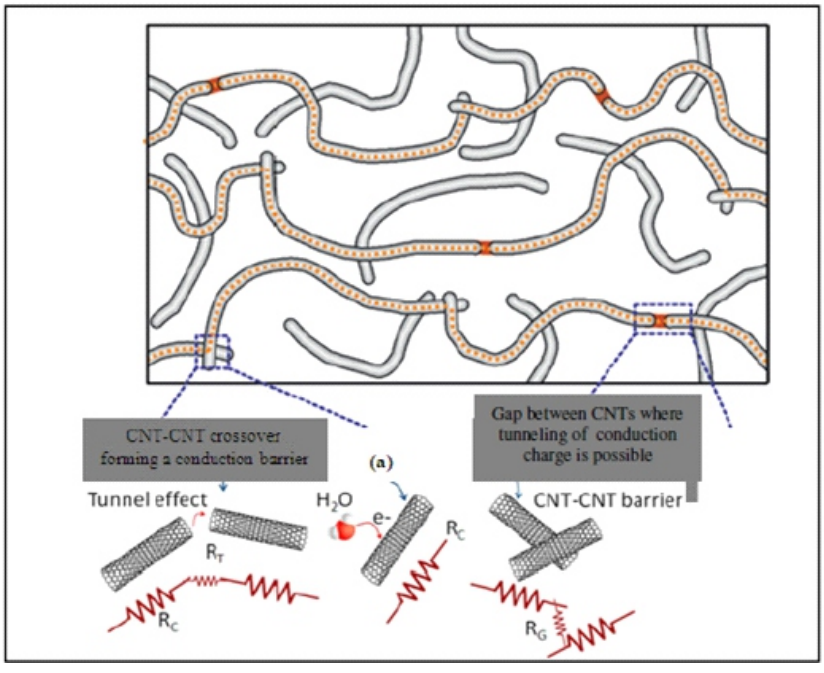

Figure 6: CNT network in (a) of CNT-polymer composite showing three types of conduction path; path resistance shown in (b), due to tunnelling at the gap between CNTs (resistance RT), due to intrinsic conductivity (resistance Rc) of CNT and due to a cross-over barrier of CNTs (resistance RG) (Kuronuma *et al.,* 2012).

CONCLUSIONS

An uncomplicated and empirical chemiresistive gas sensor for VOCs, built on Plm/MWCNTs nanocomposite film, has been successfully constructed by a fast and cost-effective wet chemistry method. The experimental results disclosed that the materials are good sensors for the measured analyte at room temperature. The proposed Plm/MWCNTs nanocomposite gas sensor provides several well-defined advantages: fast response and recovery; easy fabrication; high repeatability, room temperature operation, increased sensitivity when compared with other thin-film sensors at little gas concentration; small sample size and tunable charge transport properties as indicated in their electrical behaviour. The research findings established that Plm/MWCNTs nanocomposites has a promising future applications in gas sensing.

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