



Poly (phenyl sulfone)/graphite composite as a robust low-cost, comb-type interdigitated sensor for detection of organic solvent vapors

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Abstract

An approach for quantitative detection of organic vapors is demonstrated. Herein, an interdigitated electrode was fabricated by drop casting of graphite loaded poly(phenyl sulfone) (PPSU/Gr) film on a conductive substrate. The sensor is experimentally tested on several organic solvents including alkanes, acetates, aromatic hydrocarbons and chloroform via electrical resistance measurements and inverse gas chromatography (IGC) method. Relationships between the sensor response (as obtained by electrical resistance) and solvent properties such as polarity, acidity and partition coefficient (as obtained from IGC) are established. Moreover, the surface dispersive free energy of PPSU and PPSU/Gr were calculated according to both Dorris-Gray and Schultz equations, it is found that with the addition of graphite to PPSU the surface dispersive free energy of PPSU at 25 °C decreased from 28 to 10 mJ m⁻² (from Dorris Gray approach) and from 25 to 17 mJ m⁻² (from Schultz approach), suggesting higher interfacial area for exposed gases at PPSU/Gr sensor. Furthermore, the PPSU/Gr modified electrode exhibited high sensitivity towards chlorinated hydrocarbons. Importantly, the PPSU/Gr electrode showed good mechanical stability, low response time, and is costly-effective and disposable, rendering it a promising sensor for gases detection.

Keywords Poly (phenyl sulfone) · Graphite · Conductive composites · Solvent vapor sensor · Partition coefficient · Gas sensor

Introduction

Volatile organic solvents represent a major air pollutant [1]. These air pollutants have a significant influence on atmosphere chemistry, health and climate change. For example, toluene is neurotoxic while chloroform is carcinogenic [1]. Therefore, detection of volatile organic substances, i.e. compounds that evaporate easily at ambient conditions, is necessary from environmental and health concerns. Traditional monitoring of gases is performed using gas chromatographs (GC) which are expensive to operate and require skilled personnel. Gas sensors have been widely used to detect and quantify organic vapors [2, 3]. Typically, these sensors are made of a sensing film coated on a substrate. A multitude of coating materials for volatile organic compounds including inorganic substances, organic structures and polymer layers.

[4] Due to the large variation in polymer structures and properties, they have recently attracted great interest as recognition element for gas sensors [5–7]. However, the long-term stability and selectivity are still challenging. [6] Moreover, the design of sensor devices often relies on sophisticated procedures. Therefore, developing simple, robust and highly sensitive sensor is needed.

Polysulfone (PSU) is a polymer that exhibits an exceptional hydrolytic stability in extreme pH conditions, good electrical and adhesion properties, exceptional resistance to environmental stress and a flame retarder agent [8, 9]. Hence, PSU may provide a promising material for measurements in aqueous and gaseous media at extreme conditions. Due to the excellent properties of PSU, it has been used in electronic and medical industry. For instance, PSU has been widely used in last years in membrane separation technology and sensors field [10, 11]. PSU was also successively used in resistive humidity and gas sensors [12, 13]. They are also susceptible to incorporate biological molecules and thus are used in enzymatic and immunologic biosensors [14, 15].

Poly(phenyl sulfone) (PPSU), which is a member of the PSU family, is sold as a trademark, Radel®R. Chemical

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structure of PPSU is given in Fig. 1. PPSU is an amorphous high-tech thermoplastic polymer having better chemical, blow and temperature resistivity than PSU and polyetherimide [16]. PPSU shows resistance to steam autoclaving without loss of dimensional stability or physical properties. Therefore, PPSU has been used in the medical industry such as surgical tools. Due to its temperature resistance and useful dielectric properties, PPSU has been used in electronics [16, 17]. In this context, information about the chemical behavior and resistance of PPSU against solvent vapors is needed.

Moreover, the intrinsic conductivity of polymers can be increased by adding conductive fillers as carbon black or graphite in the polymer matrix. At a certain filler content (known as percolation threshold) a transition from insulation to conduction occurs [18]. Graphite in particular is inexpensive material and has covalent character due to the covalent bonding of carbon atoms in the structure. The electrical electricity in graphite is originated from the electron delocalization resulting from the unbound external electron of each carbon atom in its structure. [19] Benefiting from the high conductivity and possible synergistic effects of carbon and its allotropes, numerous chemosensors based on the various hydrophobic polymers doped with graphite and other conductive fillers have been investigated [20–26]. The working principle of these gas sensors is based on the percolation of gases in the polymer composite, changing its electrical resistance and possibly cause swelling up. [27] Traditional gas analyzing techniques such as gas chromatography are complex and time consuming. Gas sensors are on the other hand portable and thus can be alternative tool specially in off-laboratory applications.

Herein, a solvent vapor detection sensor based on the PPSU/Gr conductive layer is presented. Via a simple procedure, graphite powder was mixed with PPSU polymer in chloroform, forming the conductive layer. This layer was then drop casting method onto a conductive interdigitated electrode. A resistive sensor device was constructed to explore the sensing properties of PPSU/Gr against different solvent vapors such as alkanes, acetates, aromatic hydrocarbons and chloroform. The sensor performance was examined by electric resistance measurements and inverse gas chromatography (IGC). The net retention volumes, which are related to the interactions between solute and solvents, were determined by IGC. The values of the retention volumes

were used to establish a relationship between the resistance response of the sensor device and the distribution coefficient of proportional to net retention volume of solvents on the electrode. Moreover, the sensor showed excellent mechanical stability, sensitivity and low response time.

Experimental

Materials and instrumentations

PPSU polymer, Radel®R ($T_g = 220\text{ }^\circ\text{C}$) was purchased from Amoco Performance Products and was purified before use to remove impurities. Graphite powder was obtained from Fluka. To prepare homogeneous and uniform conductive film, graphite powder was sieved from about 100 μm graphite particles and the obtained smaller graphite particles of less than 45 μm are used. Studied solvents were of analytical grade and obtained from Merck. Studied samples were alkanes as octane (O), nonane (N) and decane (D); acetates as isoamyl acetate (IAA), isobutyl acetate (IBA); aromatic hydrocarbons as propyl benzene (nPB), toluene (T), iso-propyl benzene (IPB), chlorobenzene (CB) and chlorinated hydrocarbon as chloroform (Ch). They were used after purification with molecular sieves. The glass wool was treated with dimethyl dichlorosilane to close the two ends of stainless-steel column (dimensions: 1 m in length, 3.2 mm o.d.) containing PPSU/Gr covered chromosorb-W (Merck AG.Inc., acid washed and treated with dimethyldichlorosilane, pore size is 80/100 mesh) was received from Alltech Associates, Inc. and the mobile phase was high purity helium with a flowing rate in the range of 25–28 $\text{cm}^3\text{ min}^{-1}$.

The solvent vapor responses of the PPSU/Gr sensor device were measured at room temperature using HIOKI 3522–50 LCR meter with a four-point probe configuration. The retention times of the solvents on the PPSU/Gr loaded on chromosorb column were determined using HP (5890-Series II) gas chromatography. The thickness of PPSU/Gr film on the electrode was determined by Veeco Dektak XT Bruker Mechanical Profilometer. SEM analysis was examined on a Zeiss EVO LS 10 scanning electron microscope.

Designing sensor device and electrical resistance measurements

Conventional epoxy-based copper circuit board, which was made with epoxy substrate, was used to prepare the low-cost, comb-like interdigitated electrode. Firstly, the epoxy-based copper circuit board electrode was chemical etched method with $\text{HCl}:\text{H}_2\text{O}_2$ solution and then the copper lines were coated with silver in silver cyanide bath.

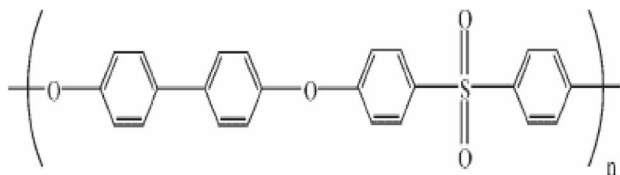


Fig. 1 Structural formula of poly (phenyl sulfone) polymer



Fig. 2 Photo image of the simple, comb-like interdigitated electrode

The prepared electrodes were washed with distilled water and acetone and dried before using (Fig. 2).

To load the PPSU/Gr conductive layer on the hand-made, comb-like interdigitated electrode (diameter = 0.8 cm), first, the required amount of PPSU polymer (0.6 g) was dissolved in chloroform and the solution was stirred to solve the whole polymer. Afterwards, graphite powder (1 g) having smaller particles (< 45 μm size) was poured into the PPSU-chloroform solution and stirred to obtain a uniform, homogeneous PPSU/Gr solution (graphite content: 60% in weight). Then, 20 μL of the PPSU/Gr solution was deposited onto the electrode under 2.0 V constant voltage, see Fig. 3. The thickness of the PPSU/Gr film was measured as 8,6 μm by mechanical profilometer. For obtaining a stable baseline resistance of PPSU/Gr sensor, the solvent vapors were evaporated at room temperature.

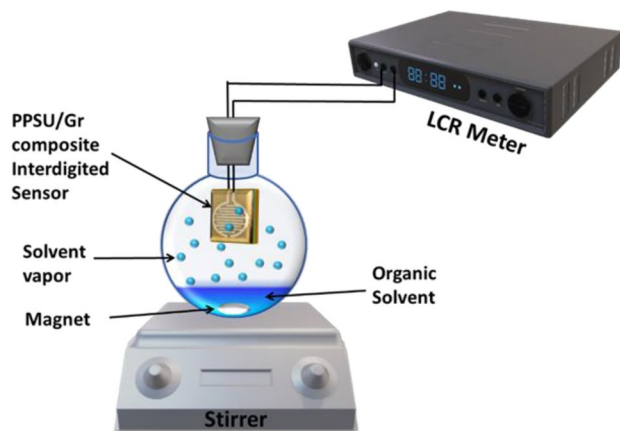


Fig. 3 The experimental set up for electrical resistance measurements using LCR meter and with the sensor device inserted in the solvent vessel

The PPSU/Gr loaded sensor device was sealed in the glass test chamber and was connected to the LCR meter and. Afterwards, 1 μl solvent was injected into the vessel, rapidly stirred to obtain a homogeneous steam, then the resistance of the sensor device towards solvent vapor was recorded continuously with the LCR meter. To remove atmospheric contaminants and remaining solvent vapors, compressed nitrogen gas was passed through the test vessel before and after measurements. The experimental set up was schemed in Fig. 3.

When a carbon/polymer composite is exposed to solvent vapors, the electrical resistance response of the polymeric composite is generally increased due to the breakage of conductive pathways as a result of swelling of the polymer. [24, 27, 28]

The relationship between the electrical resistivity of a sensor device to the solvent (R_t) and the electrical resistivity of the sensor exposure to solvent (before, R_o and after, R_t) is given as following;

$$R_r = (R_t - R_o)/(R_o) \quad (1)$$

The electrical resistivity of the polymer composite based on sensor to solvent vapors is related with the solvent partition coefficient on the polymer composite. The solvent partition coefficient on the polymer composite (K_p) is given as:

$$K_p = (C_p)/(C_v) \quad (2)$$

In Eq. 2, the vapor concentration in the polymer phase is defined as C_p , while C_v is the vapor concentration in the gas phase. [21, 29]

In the IGC technique, the solvent partition coefficient on the polymer is related to the net retention volume (V_N) of a solvent on the column which is filled with polymer. This relationship is defined as follows: [29, 30]

$$K_p = (V_N)/(w) \quad (3)$$

The weight of the solute (i.e. polymer-graphite composite) in the column is w . The net retention volume of solvent vapors on the polymer-graphite loaded on chromosorb column, V_N is determined from retention times of solvents as follows [31]:

$$V_N = Q * J * (t_R - t_A) * T/(T_r) \quad (4)$$

Surface properties of solid materials is related to the dispersive surface free energy, γ_s^d and is a specific interaction parameter that defines surface ability to behave as electron acceptor or electron donor. Dorris-Gray and Schultz equations are useful to calculate the dispersive surface free energy of the solid stationary phase from IGC measurements. [32–36] According to Dorris-Gray equation, γ_s^d is defined as;

$$\Delta G_{[CH_2]} = 2N_A a_{[CH_2]} \sqrt{\gamma_S^D \gamma_{[CH_2]}} \quad (5)$$

In Eq. 5, the adsorption energy of one methylene group, $\Delta G_{[CH_2]}$ is given by;

$$\Delta G_{[CH_2]} = -RT \ln \left[\frac{V_{N,n}}{V_{N,n+1}} \right] \quad (6)$$

The dispersive free energy changing with temperature for polyethylene is as follows:

$$\gamma[CH_2] = 35.6 + 0.058(293 - T) \quad (7)$$

where $\gamma[CH_2]$ is dispersive surface free energy of methylene groups.

According to Schultz equation, γ_S^d is given by:

$$RT \ln(V_{N,n}) = 2Na(\gamma_l^d)^{0.5}(\gamma_S^D)^{0.5} + C \quad (8)$$

In Eq. 8, the dispersive free energy of the studied n-alkane is γ_l^d , and the cross-sectional area of the n-alkane molecule is a . The $a(\gamma_l^d)^{0.5}$ data for studied n-alkane molecules are presented in Table 1.

Results and discussion

Effect of solvent properties on the sensor response: establishing solvent property-sensor response relationships

Time dependent electrical resistance changes of the PPSU/Gr composite sensor upon exposure to the solvents at saturated vapor pressure are given in Fig. 4. Some curves are not displayed in Fig. 4 for clarity.

The equilibrium responses of PPSU/Gr composite sensor to solvent vapors at saturated vapor pressures of studied solvents are presented in Fig. 5. The saturated vapor pressures

of the solvents at 25 °C were taken from previous reports. [37–41]

As shown in Fig. 4 and 5, the higher responses were obtained for chlorinated aliphatic and aromatic hydrocarbons. Although CB has a significantly low vapor pressure than that of Ch, they exhibited similar sensing response. The main difference between Ch and CB is the faster sorption–desorption behavior of Ch. Aromatic hydrocarbons showed high responses as well but with low sorption–desorption rates. On the other hand, the aliphatic esters have lower responses and sorption–desorption rates. Aliphatic hydrocarbons exhibit the lowest responses but relatively faster sorption–desorption rates. Generally, although the saturated vapor pressure of solvents is increasing, the PPSU/Gr sensor response is decreasing in aliphatic hydrocarbons and is increasing in aromatic hydrocarbons, aliphatic esters and chloroform. Increasing the volume of solvents in the test chamber containing PPSU/Gr sensor device resulted in improved sensor response in the following order: CH > CB > T > nPB > IBA > IAA > D > N > O > IPB.

The net retention volumes of solvents on PPSU/Gr adsorbed on chromosorb were calculated from net retention times using Eq. 3 and the results are presented in Table 1. The plots of $\ln(V_N)$ versus $1/T$ were given in Fig. 6 and the linear straight line was extrapolated to 25 °C to obtain V_N and K_p values at 25 °C.

Moreover, the relationship between the response of the PPSU/Gr sensor and the distribution coefficient of solvents is depicted in Fig. 7.

A correlation between the sensor responses with the partition coefficients of all studied solvents is complex. However, it is useful to make a relationship within the same class of solvents. The responses and distribution coefficient of alkanes which are nonsolvents for PPSU [40] are increasing with increasing the alkyl chain length of the alkane. The responses of aliphatic esters which are moderately poor solvent for PPSU [42] are increasing while the distribution coefficients

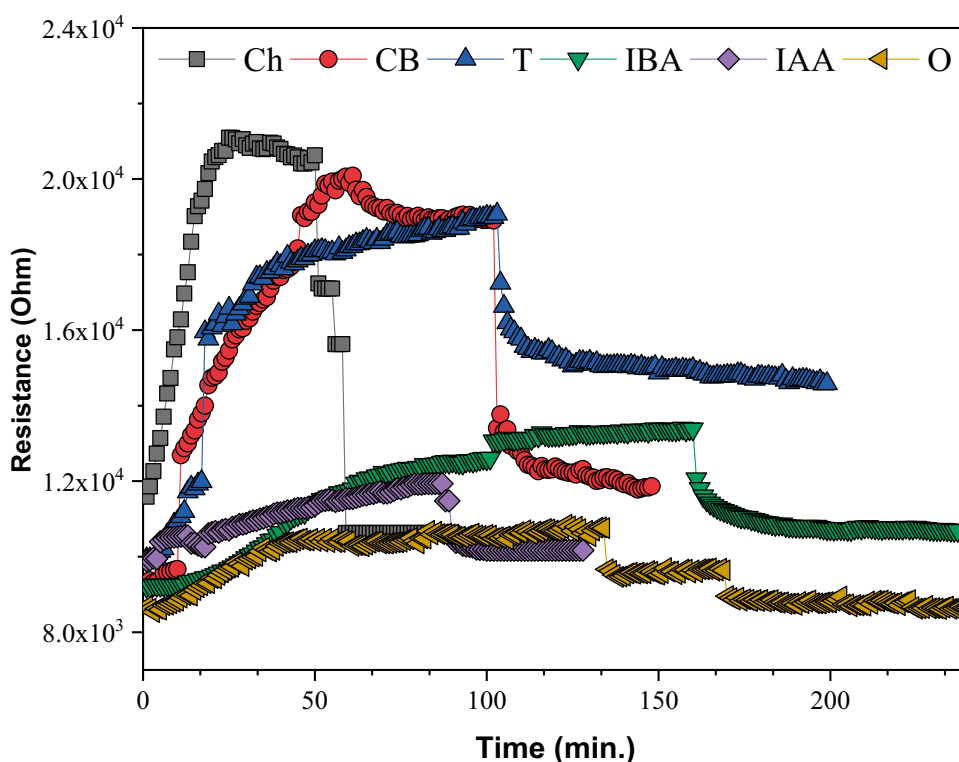
Table 1 The net retention volume, V_N for different organic solvents at different temperatures, in addition to the partition coefficients, K_p and resistivity responses, R_t on PPSU/Gr sensor at 25 °C; $a(\gamma_l^d)^{0.5}$ data are for n-alkanes

T (°C)	V_N, cm^3										
	O	N	D	IBA	IAA	T	CB	nPB	IPB	Ch	
30	6.8	23.1	45.9	10.8	19.6	15.5	25.2	33.6	21.1	8.2	
35	5.7	20.1	41.4	8.8	15.7	12.8	21.9	28.5	17.4	6.9	
40	5.1	18.3	36.3	7.4	12.7	10.6	20.1	25.0	15.3	5.9	
45	4.3	16.6	32.8	6.3	11.2	8.5	18.4	22.4	13.1	4.9	
$a(\gamma_l^d)^{0.5}$ *	2.9×10^{-18}	3.28×10^{-18}	3.63×10^{-18}	-	-	-	-	-	-	-	
V_N (25 °C)#	7.8	25.6	52.0	12.9	23.6	19.4	24.6	38.3	27.8	9.8	
K_p (25 °C)	68.1	222.5	452.4	112.5	205.2	168.4	214.3	333.2	241.4	85.3	
R_t (%)	13.9	17.51	21.9	44.5	20.5	66.1	73.0	51.7	8.1	82.0	

*Values from Refs. [40] and [41] and with $(m^2(\text{mJ}/\text{m}^2)^{0.5})$ unit

#The net retention volume of solvents at 25 °C on PPSU/Gr column was calculated by extrapolation from Fig. 6

Fig. 4 Time dependent resistance changes of PPSU/Gr composite sensor between exposure of saturated solvent vapor and nitrogen gas



are decreasing with increasing molecular weight of aliphatic esters (see Fig. 6 and 7). The results suggest that the responses are not only dependent on the type of the solvent but also on their molar volumes. The vapor pressure was not determining factor to describe the sensor response because enough time was allowed to reach to equilibrium response.

Electrostatic interactions between polymer and solvent may result in higher swelling behavior of the polymeric film. This increases the gap between the graphite particles

and thus causes an increased electrical resistance of the solvent to the polymer-graphite composite. [19, 43] This interpretation is supported with relation between chloroform/chlorobenzene and PPSU/Gr composite sensor. PPSU has a nucleophilic character due to a lone electron pair on sulfone groups and ether connections. Chloroform molecule has electrophilicity and chlorobenzene has hydrogen atoms with electron lack on the benzyl group due to the electronegative chlorine atoms. The chloroform and chlorobenzene responses of PPSU/Gr sensor device are higher which can be attributed to swelling of the polymer and increased gap between graphite particles.

Moreover, the dispersive surface free energy, γ_S^D of PPSU and PPSU/Gr at 25 °C were calculated according to Dorris Gray (Eq. 5) and Schultz (Eq. 8) approaches. By comparing the surface dispersive free energy, γ_S^D of PPSU and PPSU/Gr at 25 °C, we found that γ_S^D is lowered from 28.5 mJ m⁻² to 9.9 mJ m⁻² (calculated according to Dorris Gray approach) and from 24.8 mJ m⁻² to 17.2 mJ m⁻² (calculated according to Schultz approach) by adding graphite into PPSU. This suggests that graphite acts as a surface dispersive free energy reducing agent in the PPSU matrix.

Concentration dependency of response: calibration curve

As shown from the above results, PPSU/Gr composite film exhibits higher sensitivity against chlorinated hydrocarbons. Therefore, we investigated the use of the PPSU-based sensor to detect this important class of solvents. The relationship between

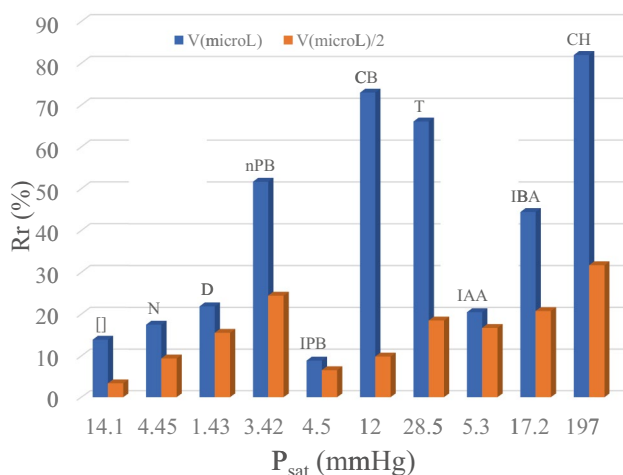
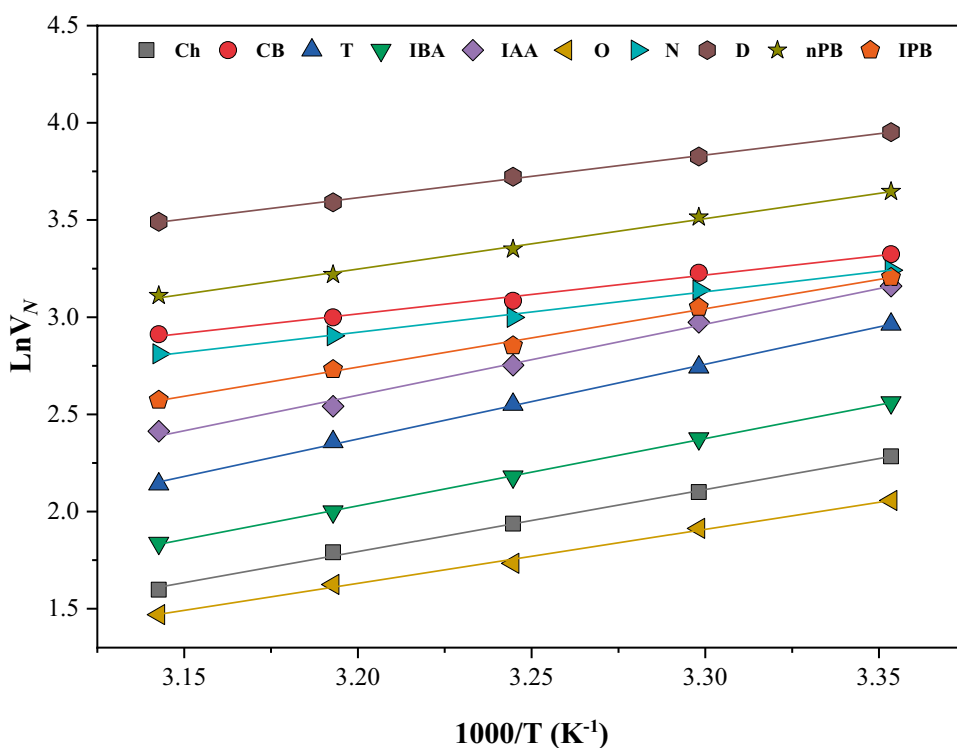


Fig. 5 Sensor responses of the PPSU/Gr composite electrode against solvent vapors measured at saturated pressure, P_{sat}

Fig. 6 The diagram of the logarithm net retention volume versus $1/T$ for different solvents on PPSU/Gr composite column



the concentration of good solvents and responses of PPSU/Gr composite sensor was demonstrated for chloroform (Fig. 8).

A linear correlation ($R_r\% = 98.76 V - 3.9454$, $R^2 = 0.979$) was obtained between the volume of exposed chloroform (between $P_{sat}/8$ and P_{sat}) and response. This linear range (calibration curve) which is obtained at the PPSU/Gr composite

electrode render this sensor promising candidate for detection of chlorinated hydrocarbons. Polyaniline (PA) and expanded graphene oxide (EGO) composite materials are exposed with various concentrations of vapours of different volatile organic compounds (VOCs) such as acetone, chloroform and carbon tetrachloride and compared with the pristine polymer, PA. The

Fig. 7 Relationship between the response of PPSU/Gr sensor and the distribution coefficient of various solvents

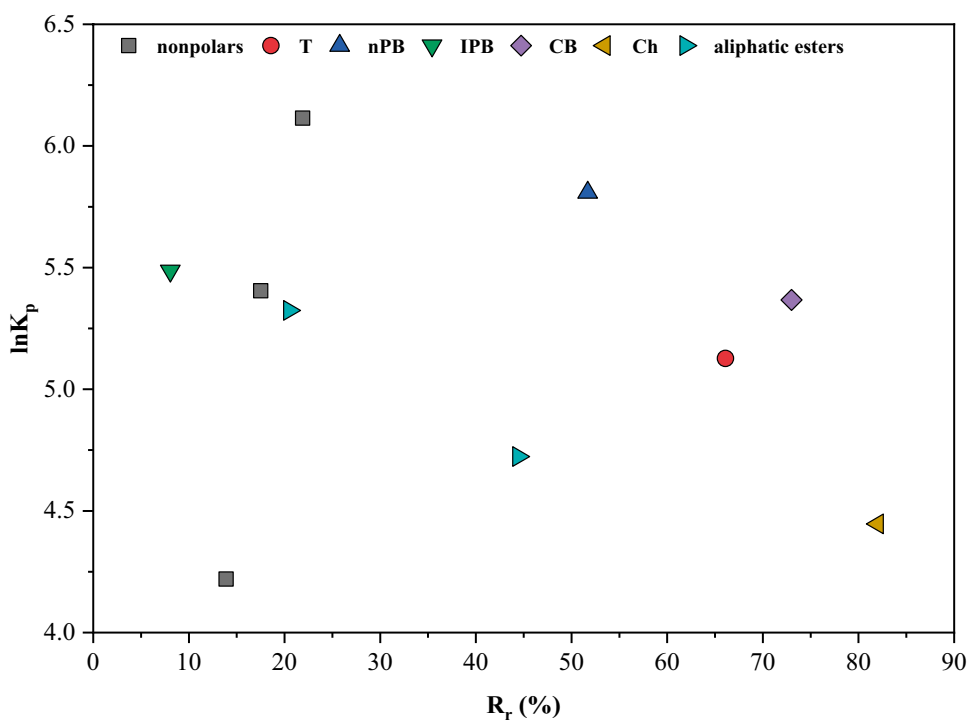
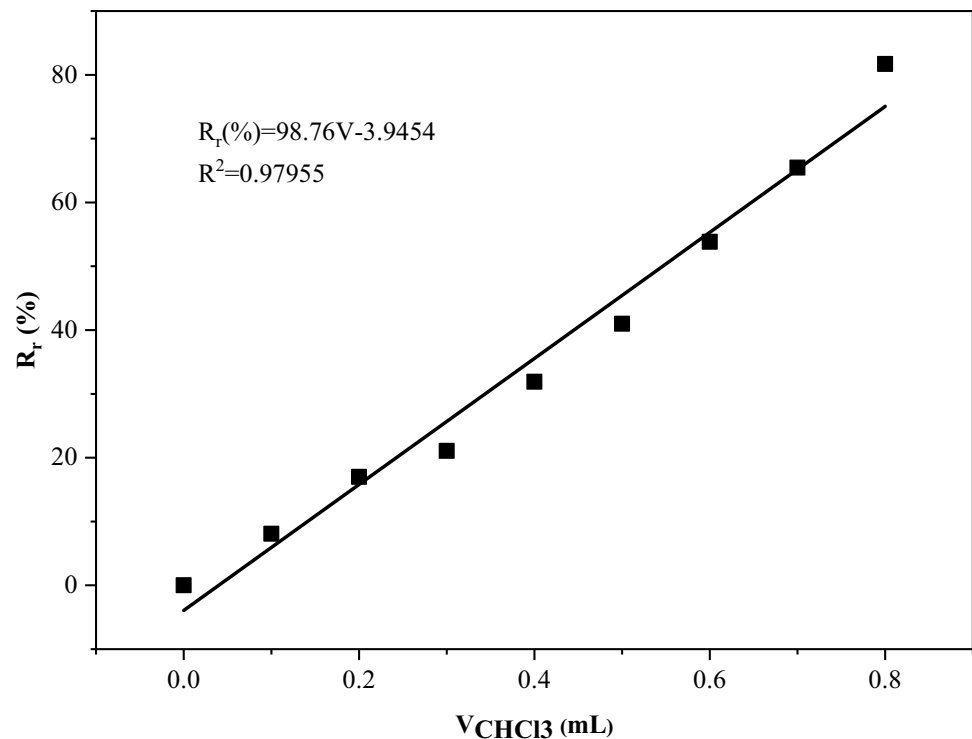


Fig. 8 Variation of PPSU/Gr sensor response as a function of exposed volume of chloroform



oxidising VOCs like acetone on exposure to pristine polymer and PA/EGO composite is found to be decrease in resistivity by hydrogen bonding with the redox sites of PA. When it is compared the chloroform sensitivity of PPSU/Gr composite with PA and PA/EGO composite, the chloroform sensitivity is found to be more in PPSU/Gr composite like PA [44].

Scanning electron microscopy (SEM) was used to investigate the surface morphology of the PPSU/Gr film on electrode. The SEM images of PPSU/Gr film on electrode were shown in Fig. 9.

As shown from the SEM images, the graphite particles are covered by PPSU and the film surface contains random distributed PPSU covered graphite particles (Fig. 9(a)). However, in spite of the random distribution of the graphite flakes in PPSU film, randomly distributed spherical zones appeared on the electrode surface. The PPSU and Gr particles have been used as the polymer matrix and conductive filler material respectively. Gr addition to PPSU matrix is improved the sensing capability and rapid response of the electrode.

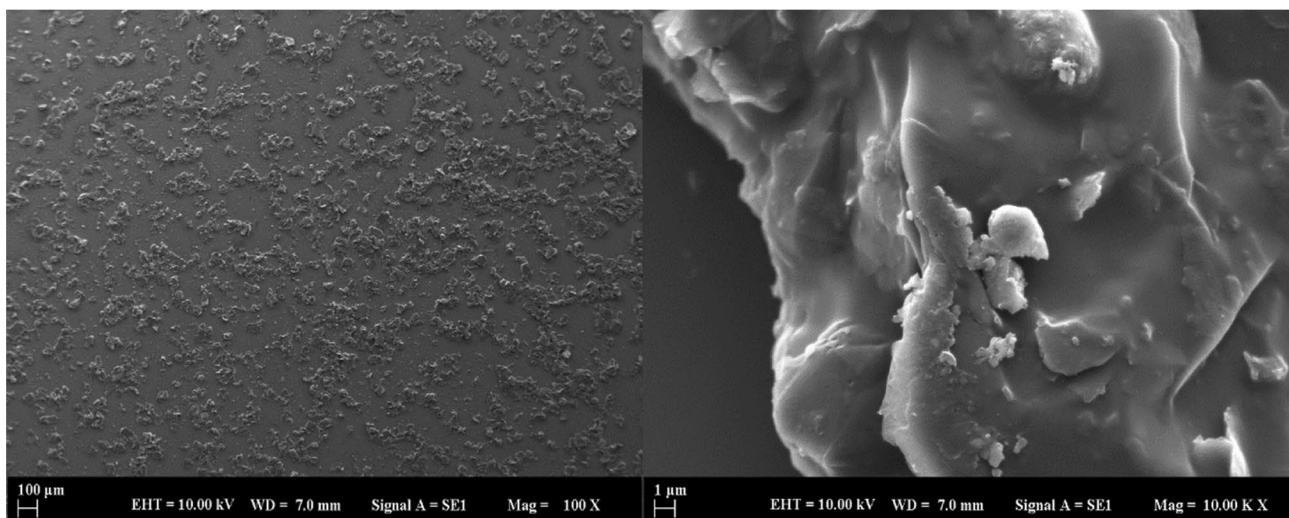


Fig. 9 The SEM images of PPSU/Gr film on electrode

Conclusions

Application of the solvent vapor concentration dependent electrical resistance measurement method on the PPSU/Gr composite film was examined and analysed. The results showed that PPSU/Gr composite film electrode is promising for sensor applications especially for environmental monitoring of air pollutants such as chlorinated hydrocarbons. The modified electrode showed good linear response towards the determination of chlorinated hydrocarbons. The addition of graphite (Gr) to PPSU polymer has reduced the surface energy of PPSU, while improving the sensitivity of PPSU sensor. The graphite-modified PPSU thin film exhibited improved sensitivity towards all tested gases, particularly towards chlorinated gases with fast response and recovery times. The reversibility of sensitive behaviour provides a potential of such composites in emerging sensing applications.

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