



Conducting poly(ether imide)–graphite composite for some solvent vapors sensing application

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ABSTRACT

The electrical resistance responses of a sensor based on graphite loaded poly(ether imide) (Ultem®1000) were measured upon exposure to various organic solvents. It was found that the responses of the composite to the solvents are drastically consistent with their relative energy difference (RED) values obtained from Hansen partial solubility parameters.

The specific retention volumes of some alkanes, aromatics and aliphatic esters similar to sensed solvents were obtained on the sensor composite in the column of a gas chromatography. It was seen that the responses of the composite to the solvent vapors vary almost linearly with the logarithm of the specific retention volumes of the corresponding solvents. The weight fraction activity coefficients at infinite dilution of the solvents were also determined at temperatures between 260 and 285 °C. It was seen that Ultem®1000/graphite composite film can be used as a sensor element to detect chloroform and chlorobenzene.

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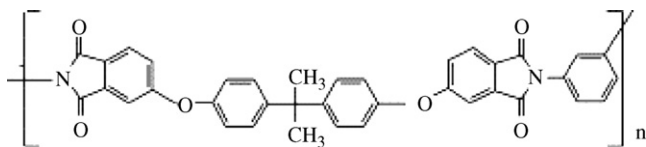
1. Introduction

During the last years, there is a great interest in using sensing devices for on-site and in situ monitoring of different chemicals. A chemical sensor basically consists of a physical transducer and a chemically selective layer. Although there are many alternatives in construction of the sensitive sensor devices by using electrochemical, thermal, mass sensitive and optical transducers, the ways in finding a highly selective recognition element for a specific analyte or analyte group are limited [1,2]. In many cases, using the multi-array sensor devices and some mathematical and statistical approximations may overcome the selectivity problems in multi-component analyses as well [3–6].

A survey of the literature reveals that polymers gained importance in the construction of sensor devices. A wide range of sensor applications of polymers was well reviewed by Adhikari and Majumdar [7]. Polymers acquired as sensing, supporting, encapsulating and immobilization materials on selective layers. A number of chemical sensors have been designed to detect solvent vapors employing carbon- or metal-loaded polymer composites based on the change of their direct current resistivity or alternative current impedance [3–15]. It was considered that the change in resistivity

or impedance is proportional to the volume and/or charge carriers mobility change of the polymer swelled with a solvent diffused and sorbed into it [8,10,13,15,16]. By swelling, the electrical resistance increases due to a breaking of the conductive pathways through the film. The degree of swelling of a particular polymer, subsequently, the response of its composite films to different solvents depends on the particular polymer–solvent interactions. On the other hand, χ , the polymer–solvent interaction parameter, and the relative energy difference (RED) of the polymer and solvent are used for predicting solubility and degree of swelling of a polymer. Inverse gas chromatography (IGC) is a versatile and very useful technique for the measurement of these parameters [17]. In this method, polymers are loaded in a chromatographic column onto an inert support and these parameters have been calculated by measuring the retention time of the solvent in the column. It seems that IGC might be used successfully in prediction of the selective polymers to the solvents in design of a chemical sensor. Ultem®1000 (Scheme 1) which is a trademark of a poly(ether imide) is an amorphous high performance thermoplastic offering high impact resistance and chemical resistance. Lower halogenated hydrocarbons such as chloroform are good solvents for it. Thus, it is candidate to prepare highly selective and sensitive sensors for halogenated hydrocarbon vapors. In this study, the DC resistance of an Ultem®1000/graphite composite film (sensor element) was measured upon exposure to a wide variety of solvent vapors. The pattern and magnitude of resistance changes of the sensor in the atmosphere of the solvent vapors were used to obtain a correlation between response and Hansen

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Scheme 1. The chemical structure of Ultem® 1000.

solubility parameters as well as retention volumes obtained by IGC measurements.

2. Theoretical background

2.1. Response of a polymer composite to a solvent

The response of the conductive particle loaded into a polymer composite to solvent vapors has been explained using percolation theory [18]. The degree of swelling of a particular polymer and the response of these composite films to different solvents depend on the particular polymer–solvent interaction [19]. The Flory–Huggins interaction parameter can also be used to predict the degree of sorption of a particular vapor into a composite [8,9].

The response of a conductive polymer composite to a solvent vapor is dependent on the partition coefficient, $K_p = C_p/C_v$ where C_p and C_v are the concentrations of the vapor in the polymer and gas phase, respectively [9,18]. The higher value of K_p means a stronger sorption of the vapor into the polymer composite and causes a significant change in the resistance of the composite material due to decreasing of conduction paths through the swelled film. The response in the electrical resistance measurement can be expressed by Eq. (1):

$$\text{response} = \frac{R_t - R_0}{R_0} \quad (1)$$

where R_t and R_0 are the resistances upon exposure and before exposure of the composite to a solvent, respectively.

2.2. Hansen solubility parameters

The Hansen partial solubility parameters being the most widely used approach on the polymer solubility prediction are called as dispersive (δ_d), polar (δ_p) and hydrogen bonding (δ_H) components of the total solubility parameter (δ_t) [20]. In this approach, a solubility sphere was defined for a polymer with the interaction radius (R_0) and the center coordinates being Hansen solubility parameter components (δ_d , δ_p and δ_H) of the polymer. The solvent is accepted as good if $RED = R_a/R_0 < 1$ or *vice versa* where R_a is calculated from the empirical Eq. (2) [20]:

$$R_a = [4(\delta_d^p - \delta_d^s)^2 + (\delta_p^p - \delta_p^s)^2 + (\delta_H^p - \delta_H^s)^2]^{1/2} \quad (2)$$

2.3. Inverse gas chromatography

The specific retention volume (V_g^0) and the weight fraction activity coefficient (Ω_1^∞) of solvents are determined experimentally from IGC measurements as follows [21–27]:

$$V_g^0 = \frac{Q(t_R - t_A)J273.2}{T_r w} \quad (3)$$

where Q is the carrier gas flow rate measured at room temperature, T_r , t_R and t_A are the retention times of the solvent and air, respectively; J is the pressure correction factor; w is the weight of the polymer in the column.

Ω_1^∞ , the weight fraction activity coefficient of solvents at infinite dilution, is defined by the following equation:

$$\ln \Omega_1^\infty = \ln \left(\frac{273.2R}{V_g^0 p_1^0 M_1} \right) - \frac{p_1^0 (B_{11} - V_1^0)}{RT} \quad (4)$$

where R is the universal gas constant; p_1^0 , M_1 , B_{11} and V_1^0 are the saturated vapor pressure, molecular weight, gaseous state second virial coefficient and molar volume of the solvent at temperature T , respectively.

The partial molar enthalpy changes accompanying to the sorption ($\Delta H_{1,s}$) and mixing (ΔH_1^∞) of the solvent at its infinite dilution in the stationary phase can be obtained by means of slopes of the corresponding plots, respectively, i.e., [21–26]

$$\Delta H_{1,s} = \frac{\partial(\ln V_g^0)}{\partial(1/T)} \quad (5)$$

$$\Delta H_1^\infty = \frac{\partial(\ln \Omega_1^\infty)}{\partial(1/T)} \quad (6)$$

3. Experimental

3.1. Materials and instrumentation

Ultem® 1000 was a product of General Electric Plastics. All solvents were purchased from Merck AG, Inc. They were analytical reagent grade and used without further purification. Dimethyl dichlorosilane treated glass wool used to plug the ends of the column was obtained from Alltech Associates, Inc. Graphite powder (<100 μm) was purchased from Fluka and sieved particles (<45 μm) were used to construct the sensing membrane.

The resistivity of the sensor was measured in a two probe configuration with a Mastech M-838 multimeter at room temperature.

A Hewlett-Packard 5890 Model, series II gas chromatography with a thermal conductivity detector was used to measure the retention time of the solvents. Data acquisition and analysis were performed by means of HP-3365 software. The column was stainless steel tubing with 3.2 mm o.d. and 1 m in length. Fourier transform infrared spectra were recorded on a PerkinElmer Spectrum One FT-IR Spectrometer. FTIR absorption spectra of the pure polymer in the native and the solvent exposed forms were obtained preferably on its thin transparent film prepared by drop casting of the polymer onto a KBr disc.

The morphological properties of surface area of the films were observed with a JOEL JSM-5410 LV scanning electron microscope.

3.2. Sensor device

The construction process of the sensor device has been given in Scheme 2. The graphite–polymer films as a sensing element were simply deposited into the cylindrical well (0.5 cm diameter) between two solvent resistant polyethylene terephthalate (PET) substrates. Each substrate with a 50 μm thermally adhesive polyethylene layer was 125 μm in thick. The construction steps of the sensor are in the following. Two parallel conductive lines were casted onto the adhesive layer of the first PET substrate by using a silver conductive ink (step 1). Then, the second hole-punctured PET substrate was prepared (step 2) and thermally stickled onto the first one giving a cylindrical exposing volume with two electrical contacts for composite film (step 3). The polymer was first dissolved in chloroform then the required amount of graphite powder (<45 μm) was added to the solution to obtain the graphite content of 60% (w/w) in solid state and thoroughly mixed. Approximately, 5 μL of homogenous suspension was injected into the well (step 4). The solvent was evaporated at room temperature until a stable baseline resistance around 500 Ω with a maximum deviation of

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