



Research Article

THERMODYNAMICS OF A CHALCONE-BASED POLYMER BY INVERSE GAS CHROMATOGRAPHY METHOD

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ABSTRACT

In this study, an inverse gas chromatography (IGC) study of thermodynamic interactions of a chalcone modified poly (styrene) based polymer (PVBC-DMAC) with some solvents was presented. For this purpose, the retention data of carefully selected polar and non-polar solvents on PVBC-DMAC was obtained by IGC instrument at temperatures between 358.2 K and 383.2 K. Then, the polymer-solvent interaction parameters for Flory-Huggins theory, χ_{12}^{∞} and the equation of state theory, χ_{12}^* , the effective exchange energy parameters, X_{eff} , the weight fraction activity coefficient, Ω_1^{∞} , and also the partial molar heat of mixing at infinite dilution of solvent, $\Delta\bar{H}_1^{\infty}$, the molar heat of vaporization of solvent, $\Delta\bar{H}_v$, and the partial molar heat of sorption of solvent, $\Delta\bar{H}_1^s$ were calculated. The obtained results were discussed.

Keywords: Inverse gas chromatography, thermodynamics, poly (styrene) based polymer.

1. INTRODUCTION

Photosensitive polymers having a photocrosslinkable groups have received increasing attention in recent years largely. Polymers containing chalcone groups (α,β -unsaturated carbonyl groups, eg., cinnamoyl groups) either in the main chain or as pendant group can be crosslinked by irradiation with UV light or an electron beam, and such polymers are widely used as photoresists in the field of printing technology [1], microlithography [2], non-linear optical materials [3], liquid crystals [4], holographic head-up display [5], energy exchange materials [6] etc. Polymers with good solubility, thermal stability, the ability to form films, high photosensitivity, are of great importance in practical application such as photoresist.

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Knowledge of thermodynamics of polymer solutions and their blends are essential to finding optimum conditions for the design of many processes and products in industrial and laboratory applications [7-9]. Especially, the thermodynamic quantities such as Flory–Huggins interaction parameters χ_{12}^{∞} and the weight fraction activity coefficients Ω_1^{∞} can be used for characterization of different polymeric systems [10]. They are a useful tool to predict enough miscibility or solubility between a solvent and a polymer/polymer-polymer blends.

Inverse gas chromatography (IGC) method which was developed by Smidsord and Guillet [11] has been commonly applied to characterize non-volatile materials, including polymeric systems. In the IGC method, the stationary phase of the chromatographic column is an unknown polymeric material under study, in contrast to traditional gas chromatography. Many interaction and thermodynamic characteristics of polymers, such as χ_{12}^{∞} , Ω_1^{∞} , the partial molar heat of mixing at infinite dilution of the solvent $\Delta\bar{H}_1^{\infty}$, molar heat of vaporization of solvent $\Delta\bar{H}_v$ and the partial molar heat of sorption of the solvent $\Delta\bar{H}_1^s$ can be determined by IGC, over a wide temperature range [10,12-22]. The method is simple, rapid and efficient compared with other techniques applied for physicochemical characterization [23-24]. Moreover, as a small amount of the solvent (probe) and polymer are used for IGC measurements, the method is economical.

The objective of this work was to perform a thermodynamic characterization of a chalcone modified poly (styrene) based polymer (PVBC-DMAC) [25] by the aid of the IGC method. Firstly, we have investigated the interactions of PVBC-DMAC with ethyl acetate (EA), ethyl benzene (EB), n-butyl acetate (nBAC), iso-butyl acetate (iBAC), n-propylbenzene (nPb), iso-propylbenzene (iPB), toluene (T), n-heptan (Hp), n-octane (O), n-nonane (N), n-decane (D), n-undecane (UD), n-dodecane (DD) and n-tridecane (TD) probes in the temperature range from 358.2 up to 383.2 K at infinite dilution. Finally, we have calculated some thermodynamic parameters for PVBC-DMAC.

2. DETERMINATION OF THERMODYNAMIC PARAMETERS BY IGC METHOD

To perform IGC measurements, the polymer under investigation is filled into the chromatographic column as stationary phase and selected solvents (probes) are injected into the column to obtain the retention data. In the method, there's a relation between the polymer-probe interactions and the specific retention volumes V_g^0 .

V_g^0 of the probe is determined experimentally by using IGC measurements as given by Equation (1) [11,26-27];

$$V_g^0 = Q(t_R - t_A) \cdot J \cdot 273.2 / (T_r \cdot w) \quad (1)$$

Where Q is flow rate of the carrier gas measured at room temperature T_r ; t_R and t_A are retention times of the probe and air, respectively; J is the pressure correction factor; w is the weight of the polymer in the IGC column. Based on theories of Flory-Huggins and equation-of-state interaction parameters, χ_{12}^{∞} and χ_{12}^* are determined from the following Equations (2) and (3), respectively [28, 29]:

$$\chi_{12}^{\infty} = \ln \left(\frac{273.2 R v_2}{p_1^0 V_g^0 V_1^0} \right) - 1 - \frac{p_1^0 (B_{11} - V_1^0)}{RT} \quad (2)$$

$$\chi_{12}^* = \ln \left(\frac{273.2 R v_2^*}{p_1^0 V_g^0 V_1^*} \right) - 1 - \frac{p_1^0 (B_{11} - V_1^0)}{RT} \quad (3)$$

where R is the universal gas constant; p_1^0 and B_{11} are saturated vapor pressure, gaseous state second virial coefficient; V_1^0 is the molar volume of the solvent at temperature T ; v_2 and v_2^* are specific volume and specific hardcore volume of the polymer in the column, respectively. V_1^* is the molar hardcore volume of the solvent. The effective exchange energy parameter, X_{eff} , in the equation of state theory is equal to [26, 27]:

$$RT\chi_{12}^* = p_1^*V_1^* \left\{ 3T_{1r} \ln \left[\left(v_{1r}^{1/3} - 1 \right) / \left(v_{2r}^{1/3} - 1 \right) \right] + v_{1r}^{-1} - v_{2r}^{-1} + X_{eff} / P_1^* v_{2r} \right\} \quad (4)$$

Where p_1^* is characteristic pressure, v_{1r} and v_{2r} are reduced volume of the solvent and the polymer, respectively. T_{1r} is a reduced temperature of the solvent. The partial molar heat of sorption, $\Delta\bar{H}_1^s$ of the solvent sorbed by the solute is given by the equation:

$$\Delta\bar{H}_1^s = -R \left(\frac{\partial(\ln V_g^0)}{\partial(1/T)} \right) \quad (5)$$

The partial molar heat of mixing, $\Delta\bar{H}_1^{\infty}$, is obtained from Equation (6):

$$\Delta\bar{H}_1^{\infty} = R \left(\frac{\partial(\ln \Omega_1^{\infty})}{\partial(1/T)} \right) \quad (6)$$

Ω_1^{∞} is the weight fraction activity coefficient of solvent at infinite dilution, is expressed as follows:

$$\ln \Omega_1^{\infty} = \ln(273.2R/V_g^0 p_1^0 M_1) - p_1^0 (B_{11} - V_1^0) / RT \quad (7)$$

where, M_1 is the molecular weight of probe. The molar heat of vaporisation, $\Delta\bar{H}_v$, of the probe is related to $\Delta\bar{H}_1^s$ and $\Delta\bar{H}_1^{\infty}$ as follows:

$$\Delta\bar{H}_v = \Delta\bar{H}_1^{\infty} - \Delta\bar{H}_1^s \quad (8)$$

3. EXPERIMENTAL

3.1. Materials and Instrumentation

All solvents and other chemicals were purchased from Merck AG Inc. Chromosorb-W (AW-DMCS-treated, 80/100 mesh) used as the solid support was supplied from Merck AG Inc. Silanized glass wool used to plug the ends of the chromatography column was obtained from Alltech Associates, Inc. The IGC experiments were carried out on an Agilent Technologies 6890N model gas chromatography with thermal conductivity (TCD) detector. The chromatography column made of stainless steel tubing (1 m length x 3.2 mm outside diameter) was obtained from Alltech Associates, Inc.

3.2. Preparation of the column

PVBC-DMAC was prepared as described in the literature [25]. The technique of solvent evaporation was employed for coating support material with the polymer a weighed amount of the solid support material was added to the solution of PVBC-DMAC in DMSO and solvent was slowly evaporated by stirring. The weight of polymeric phase was 0.1389 g. The amount of coated the polymer on the support was determined as 10.68 % by calcination. A 1 μ L Hamilton syringe was used to inject a trace amount of probe into the column. Before starting the IGC measurements, the prepared column was conditioned at 423.2 K for 24 h.

4. RESULTS AND DISCUSSION

The synthesis procedure of PVBC-DMAC and a study on determination its surface properties by IGC method are available in literature [25]. The specific retention volume, V_g^0 is used for physicochemical characterization of polymer-probe systems by IGC experiments. The values of V_g^0 of polar solvents as EA, EB, nBac, iBac, nPB, iPB, T and non-polar solvents as Hp, O, N, D, UD, DD and TD on PVBC-DMAC were obtained from IGC measurements at temperatures between 358.2 and 383.2 K using Equation (1). The glass transition temperature value of PVBC-DMAC was found to be 335.2 K in ref. [25]. Sorption properties of a polymer in a liquid state can

be studied at temperatures higher than T_g in the equilibrium sorption region to obtain useful information about thermodynamics related to interactions between polymer and a solvent. The linearity in the retention diagram shows the thermodynamic equilibrium. Therefore the thermodynamic properties of PVBC-DMAC were investigated at the temperatures between 358.2 and 383.2 K. Figures 1 and 2 show the variation of the specific retention volumes of non-polar and polar solvents in the studied temperature range.

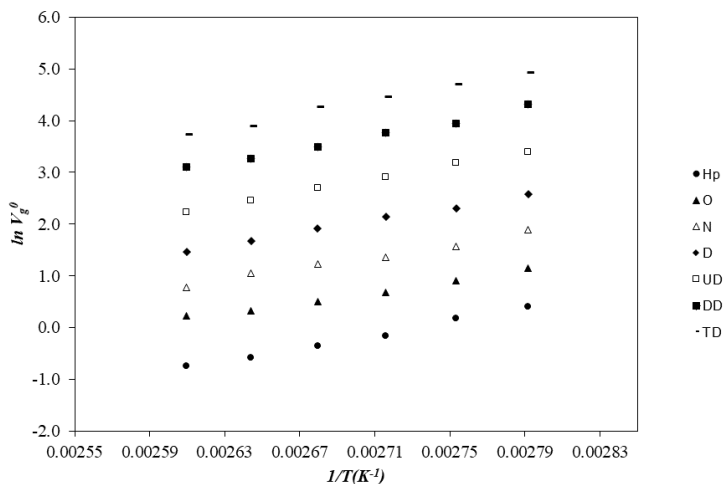


Figure 1. $\ln V_g^0 - 1/T$ diagram of the non-polar solvents on PVBC-DMAC.

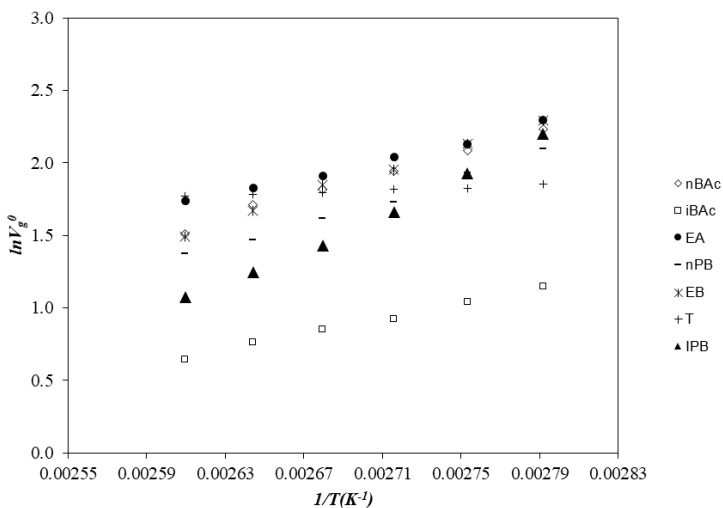


Figure 2. $\ln V_g^0 - 1/T$ diagram of the polar solvents on PVBC-DMAC.

As shown in Fig.1 and Fig.2 it was observed that the values of $\ln V_g^0$ for all probes decreased with increasing temperature

The polymer-solvent interaction parameters, χ_{12}^∞ and χ_{12}^* calculated from Equations (2) and (3) are presented in Tables 1 and 2, respectively.

Table 1. Flory–Huggins polymer-solvent interaction parameters, χ_{12}^{∞} , of PVBC-DMAC with selected probes at temperature between 358.2 and 383.2 K.

T(K)	Hp	O	N	D	UD	DD	TD	EB	EA	nBAc	iBAc	nPB	iPB	T
358.2	4.33	4.39	4.44	4.56	4.55	4.43	4.64	3.76	2.09	3.51	4.24	4.62	4.22	3.31
363.2	4.40	4.47	4.57	4.61	4.51	4.53	4.58	3.74	2.10	3.47	4.17	4.59	4.31	3.17
368.2	4.60	4.51	4.60	4.58	4.56	4.47	4.55	3.74	2.05	3.43	4.12	4.60	4.39	3.03
373.2	4.65	4.54	4.55	4.61	4.55	4.51	4.49	3.68	2.04	3.39	4.03	4.53	4.45	2.90
378.2	4.73	4.55	4.55	4.66	4.59	4.50	4.60	3.69	1.99	3.33	3.96	4.50	4.46	2.77
383.2	4.77	4.50	4.66	4.67	4.61	4.45	4.52	3.72	1.95	3.37	3.92	4.43	4.47	2.65

Table 2. The equation of state polymer-solvent interaction parameter, χ_{12}^* , of PVBC-DMAC with selected probes at temperature between 358.2 and 383.2 K.

T(K)	Hp	O	N	D	UD	DD	TD	EB	EA	nBAc	iBAc	nPB	iPB	T
358.2	3.69	4.51	4.56	4.67	4.66	4.53	4.74	3.87	2.25	3.63	4.37	4.72	4.32	3.43
363.2	4.54	4.59	4.68	4.72	4.62	4.63	4.67	3.85	2.27	3.59	4.31	4.69	4.41	3.30
368.2	4.74	4.64	4.71	4.68	4.66	4.56	4.64	3.85	2.22	3.56	4.25	4.70	4.49	3.15
373.2	4.80	4.66	4.66	4.71	4.65	4.61	4.58	3.79	2.22	3.52	4.16	4.63	4.55	3.03
378.2	4.88	4.68	4.66	4.76	4.68	4.59	4.68	3.81	2.17	3.46	4.10	4.60	4.56	2.90
383.2	4.92	4.63	4.77	4.77	4.70	4.53	4.60	3.83	2.13	3.50	4.06	4.53	4.57	2.78

The values $\chi_{12}^{\infty} < 0.5$ suggest strong intermolecular interactions between polymer chains and solvent molecules, whereas the values $\chi_{12}^{\infty} > 0.5$ indicate poor interactions in dilute polymer solutions. As can be seen in Table 1 and 2, the values of χ_{12}^{∞} and χ_{12}^* suggest that all the studied solvents are poor for PVBC-DMAC. According to the values of χ_{12}^{∞} , the solubility of polymer in TD, EB, EA, nBAc, iBAc, nPB and T are endothermic, however Hp, O, N, D, UD and iPB are exothermic.

The values of X_{eff} were determined by Equation (4) and presented in Table 3. X_{eff} quantifies the change in contact energy when a polymer segment in the vicinity of another polymer segment is replaced by a solvent molecule [26, 27]. The higher values of X_{eff} represent poor solubility while the lower values of X_{eff} represent good solubility due to specific interactions between solvents and the polymer.

Table 3. Effective exchange energy parameters of the solvents, X_{eff} (j/cm^3), of PVBC-DMAC with selected probes at temperature between 358.2 and 383.2 K.

T(K)	Hp	O	N	D	UD	DD	TD
358.2	123.48	111.95	102.35	96.40	88.45	79.59	78.10
363.2	127.24	115.60	106.78	98.65	88.72	82.52	77.89
368.2	135.18	118.38	108.82	99.00	90.77	82.21	78.24
373.2	138.59	120.45	108.93	100.91	91.61	84.08	78.11
378.2	142.98	122.41	110.27	103.25	93.43	84.68	81.03
383.2	146.03	122.39	114.48	104.81	94.89	84.60	80.42
T(K)	EB	EA	nBAc	iBAc	nPB	iPB	T
358.2	129.82	81.49	109.31	122.69	136.76	127.70	132.45
363.2	130.69	83.26	109.31	122.04	137.64	132.11	128.39
368.2	132.46	81.47	109.46	121.78	139.83	136.49	123.58
373.2	131.82	82.03	109.06	120.26	139.36	140.14	119.47
378.2	133.97	80.29	108.31	119.43	140.09	142.28	115.06
383.2	136.50	79.15	111.00	119.56	139.35	144.27	110.50

The parameter Ω_1^∞ derived from fundamental relations of physicochemistry does not include any uncertainty coming from theoretical assumptions. According to Guillet [28], when Ω_1^∞ is lower than 5 the solvent is characterized as a good solvent for the polymer, while a value higher than 10 designates a poor solvent. The values between 5 and 10 indicate moderately good solubility. The values of Ω_1^∞ the studied solvents at infinite dilution were calculated from Equation (7) and are presented in Table 4. The values of Ω_1^∞ found in this study suggest that all the studied solvents are poor solvents for PVBC-DMAC.

Table 4. The weight fraction activity coefficients of the solvents, Ω_1^∞ , of PVBC-DMAC with selected probes at temperature between 358.2 and 383.2 K.

T(K)	Hp	O	N	D	UD	DD	TD
358.2	234.09	229.03	226.10	240.64	224.17	187.46	219.23
363.2	250.89	248.05	256.20	251.73	215.38	207.13	205.04
368.2	306.15	259.91	263.41	242.40	225.17	193.61	198.16
373.2	322.76	265.46	251.13	249.76	222.46	202.27	186.85
378.2	350.21	269.93	251.39	261.80	229.97	198.33	207.79
383.2	364.08	256.56	280.30	265.45	233.74	188.11	191.29
T(K)	EB	EA	nBAc	iBAc	nPB	iPB	T
358.2	108.50	21.06	81.51	180.02	249.89	165.52	71.65
363.2	106.17	21.47	78.49	168.29	242.49	180.25	62.81
368.2	106.50	20.38	76.02	159.58	245.51	195.67	54.35
373.2	100.29	20.29	72.56	145.86	228.77	207.40	47.98
378.2	101.73	19.32	68.71	136.48	221.70	209.79	42.25
383.2	104.13	18.62	71.54	131.57	205.72	211.19	37.19

Table 5. The values of, $\Delta\overline{H}_1^s$ (kcal/mol), $\Delta\overline{H}_1^\infty$ (kcal/mol), $\Delta\overline{H}_v$ (kcal/mol) [29] of the studied solvents.

Solvents	$\Delta\overline{H}_1^\infty$	$\Delta\overline{H}_1^s$	$\Delta\overline{H}_v$	$\Delta\overline{H}_{vl}$
Hp	-5.1	-12.9	7.8	7.6
O	-1.3	-10.2	8.8	8.2
N	-1.5	-11.4	9.9	8.8
D	-0.9	-11.9	10.9	9.4
UD	-0.6	-12.7	12.1	9.9
DD	0.1	-13.1	13.2	10.4
TD	1.1	-13.3	14.4	10.9
EB	0.6	-8.5	9.1	8.5
EA	1.4	-5.9	7.4	7.7
nBAc	1.7	-7.6	9.3	8.6
iBAc	3.6	-5.3	8.9	8.7
nPB	2.0	-7.9	10.0	9.1
iPB	-2.7	-12.3	9.6	9.0
T	7.2	-0.9	8.0	7.9

The values of $\Delta\overline{H}_v$ were obtained from Equation 8 and were also compared to the literature values, $\Delta\overline{H}_{vl}$. The obtained results were presented in Table 5. The agreement is good for the solvents that boiling temperature is close to the average of the studied column temperatures. This comparison indicates that the reliability of the thermodynamic characteristics obtained in this study is considerable.

The analysis of $\Delta\overline{H}_1^\infty$, $\Delta\overline{H}_1^s$ and $\Delta\overline{H}_v$ parameters is useful for understanding sorption mechanism. $\Delta\overline{H}_1^s$ values of sorption of the solvents on PVBC-DMAC were obtained from the slope of a plot of $\ln V_g^0$ versus $1/T$ in the 358.2-383.2 K range (Table 5). The partial molar enthalpy of mixing $\Delta\overline{H}_1^\infty$ describes solvent-polymer interactions in the condensed phase. $\Delta\overline{H}_1^\infty$ values were obtained from the slopes of the plots of $\ln\Omega_1^\infty$ versus $1/T$ in the studied temperature ranges using Equation (6). According to the values of $\Delta\overline{H}_1^\infty$, the solubility of polymer in TD, EB, EA, nBac, iBac, nPB and T are endothermic, however Hp, O, N, D, UD and iPB are exothermic.

5. CONCLUSIONS

IGC method at infinite dilution was utilized for determination of the thermodynamic properties (the Flory Huggins and equation of state polymer-solvent interaction parameters, effective energy parameters and the partial molar heat of mixing) of PVBC-DMAC. The values of interaction parameters found in this study suggest that the studied solvents are poor for the polymer. According to the values of χ_{12}^∞ and $\Delta\overline{H}_1^\infty$, the solubility of polymer in TD, EB, EA, nBac, iBac, nPB, T are endothermic, however Hp, O, N, D, UD, iPB are exothermic.

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