DETERMINATION OF GLASS TRANSITION TEMPERATURE AND SURFACE PROPERTIES OF NOVEL CHALCONE MODIFIED POLY (STYRENE) BASED POLYMER

by

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The novel chalcone modified poly (styrene) based polymer (PVBC-DMAC) has been prepared and characterized. By using the inverse gas chromatography method, we determined glass transition temperature and surface properties of PVBC-DMAC. The obtained glass transition temperature, T_g, *is a good agree with the one obtained by differential scanning calorimetry. Also considering some isomeric alcohol and acetate solvents in the temperature range from 303.2 K to 353.2 K, we investigated the polymer selectivity. The parameters* K_A *(acidic) and* K_D *(basic) of the PVBC-DMAC surface were calculated. The obtained* K_A, K_D *values reflected that PVBC-DMAC surface has a basic character.*

Key words: *inverse gas chromatography, glass transition temperature,*
chalcone modified poly (styrene) based polymer,
surface properties, isomer solvent seperation

Introduction

The photosensitive polymer containing chalcone (α-, β-unsaturated carbonyl) groups either in the backbone or as a side chain can be cross-linked by (UV light or an electron beam) irradiation. These polymers are important for several practical application due to possessing good thermal stability and solubility, the ability to form films, high photosensitivity, resistance towards solvents after crosslinking and good flame retardance. They are being used as photoresists in a wide variety of application [1-6]. Inverse gas chromatography (IGC) that is a simple, reliable and inexpensive method, is a widely-used method to examine the thermodynamic and surface properties of polymers and their blends, over a wide temperature range [7, 8]. The these properties that can be determined for polymeric systems include polymer/solvent and polymer/polymer interaction parameters, solubility parameters, surface energies and Lewis acid-base parameters, molar heats of mixing and sorption, diffusion kinetics and thermal transitions [9-20].

In this paper, we synthesized and characterized the novel chalcone modified poly (styrene) based polymer (PVBC-DMAC). Then, glass transition temperature, T_g, and the se-

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lectivity of PVBC-DMAC were examined using some isomeric alcohol and acetate solvents through IGC method at temperatures between 303.2 and 353.2 K. The surface free energy and acid-base characteristics of PVBC-DMAC were determined through measurements of net retention volumes of the polar and non-polar solvents and by use of adsorption on principle in IGC. The retentions of ten organic compounds of different chemical nature and polarity were measured in the temperature range between 303.2 and 328.2 K.

Theory

Based on the retention time for a selected solvent, the polymer-solvent interactions are determined. A net retention volume, V_N, is presented the following form:

$$V_N = \frac{QJ\left(t_R - t_A\right)T}{T_f} \tag{1}$$

where Q is carrier gas flow rate measured at room temperature T_f [K], J – the correction factor, t_R – the retention times of the solvent, t_A – the retention times of air, and T [K] – the column temperature [21, 22].

Interactions between adsorbent and adsorbate molecules can be specific, γ_S^S, and dispersive, γ_S^D, [23]. They are given as components of the free surface energy, γ_S, of the adsorbent by the following form:

$$\gamma_S = \gamma_S^D + \gamma_S^S \tag{2}$$

Dorris-Gray [24] and Hamieh and Schultz [25] methods are used at the obtaining of surface dispersive energy. According to the Dorris-Gray method, γ_S^D is defined:

$$\Delta G_{A[\mathrm{CH_2}]} = 2N_A a_{[\mathrm{CH_2}]}\sqrt{\gamma_S^D \gamma_{L[\mathrm{CH_2}]}} \tag{3}$$

where $\Delta G_{A[\mathrm{CH_2}]}$ is the adsorption free energy of one methylene group, N_A – the Avogadro constant, $a_{[\mathrm{CH_2}]}$ – the one methylene group's (CH$_2$) cross-sectional area, and $\gamma_{L[\mathrm{CH_2}]}$ – the surface free energy of adsorption of one methylene group.

The $\Delta G_{A[\mathrm{CH_2}]}$ is calculated:

$$\Delta G_{A[\mathrm{CH_2}]} = -\mathrm{R}T\ln\left(\frac{V_{N,n}}{V_{N,n+1}}\right) \tag{4}$$

where R is the universal gas constant, $V_{N,n}$ and $V_{N,n+1}$ are the volumes of alkanes with *n* and (*n*+1) carbon atoms in their chain, respectively. The graph of the $\mathrm{R}T\ln V_N$ values of *n*-alkanes against the number of carbon atoms of *n*-alkanes is a linear and the slope of the linear graph is equal to $\Delta G_{A[\mathrm{CH_2}]}$. Hence considering the experimentally determined the values of $V_{N,n}$ and $V_{N,n+1}$, eqs. (3) and (4), it is possible to calculate the dispersion component of the surface free energy, γ_S^D.

The free energy of adsorption, ΔG_A, is associated with V_N:

$$\Delta G_A = -\mathrm{R}T\ln\left(V_N\right) + K \tag{5}$$

where K is a constant for the studied column [18].

According to Schultz method, γ_L^D is associated with V_N:

$$-\Delta G_A = \mathrm{R}T\ln\left(V_N\right) = 2Na\left(\gamma_S^D\right)^{0.5}\left(\gamma_L^D\right)^{0.5} + K'' \tag{6}$$

where γ_L^D is the dispersive component of the surface free energy of the adsorbent [26]. For a series of *n*-alkane solvents, the slope of a graph of $\mathrm{R}T\ln V_N - a(\gamma_L^D)^{0.5}$ is equal to $2N(\gamma_S^D)^{0.5}$. Values of $a(\gamma_L^D)^{0.5}$ are obtained from [18, 23, 24].

The values of $a(\gamma_L^D)^{0.5}$ for n-alkanes are shown in the tab. 1 [18, 26, 27]. The plot of $\mathrm{R}T\ln V_N$ as a function of $a(\gamma_L^D)^{0.5}$ for a series of *n*-alkanes is linear of which the gradient gives the specific component of the free energy.

Table 1. The values of $a(\gamma_L^D)^{0.5}$ for the studied *n*-alkane solvents

Solvent	a [$\cdot 10^{-10}$m^2]	γ_L^D [mJm^{-2}]	$a(\gamma_L^D)^{0.5}$ [m^2(mJm^{-2})$^{0.5}$]
Hx	51.5	18.4	$2.21\cdot 10^{-18}$
Hp	57.0	20.3	$2.57\cdot 10^{-18}$
O	62.8	21.3	$2.90\cdot 10^{-18}$
N	69.0	22.7	$3.28\cdot 10^{-18}$
D	75.0	23.4	$3.63\cdot 10^{-18}$

On the diagram, polar solvent points do not lie on the alkane line and the vertical distance from the *n*-alkane reference line to the specific free energy of adsorption, $-\Delta G_A^S$. The procedure can be expressed:

$$-\Delta G_A^S = \mathrm{R}T\ln\left(\frac{V_{N,n}}{V_{N,\mathrm{ref}}}\right) \tag{7}$$

where $V_{N,\mathrm{ref}}$ and $V_{N,n}$ are the retention volume for the *n*-alkanes reference line and the retention volume for the polar solvent, respectively.

Values of $a(\gamma_L^D)^{0.5}$, the Gutmann's modified acceptor number, AN^*, and donor number, DN, of the polar solvent studied in this work are shown tab. 2 [28, 29].

Table 2. The values of $a(\gamma_L^D)^{0.5}$, *DN* and *AN for the studied polar solvents**

Solvent	a [$\cdot 10^{-10}$m^2]	γ_L^D [mJm^{-2}]	$a(\gamma_L^D)^{0.5}$ [m^2(mJm^{-2})$^{0.5}$]	AN^* [kJmol^{-1}]	DN [kJmol^{-1}]
DCM	31.5	27.6	$1.65\cdot 10^{-18}$	16.4	0.0
TCM	44.0	25.9	$2.24\cdot 10^{-18}$	22.7	0.0
THF	45.0	22.5	$2.13\cdot 10^{-18}$	2.1	84.0
Ace	42.5	16.5	$1.73\cdot 10^{-18}$	10.5	71.4
EA	48.0	19.6	$2.13\cdot 10^{-18}$	6.3	71.8

The adsorption of a polar solvent on the surface of investigated adsorbent causes a change in the enthalpy and the entropy of the system. The specific component of the surface free energy is given:

$$\Delta G_A^S = \Delta H_A^S - T\Delta S_A^S \tag{8}$$

where ΔH_A^S and ΔS_A^S are the adsorption enthalpy and adsorption entropy for Lewis acid-base interactions, respectively. Then by plotting $\Delta G_A^S/T$ values of the polar molecules as a function of T^{-1}, ΔH_A^S, and ΔS_A^S are found for each polar solvent by using equation:

$$\frac{\Delta G_A^S}{T} = \frac{\Delta H_A^S}{T} - \Delta S_A^S \tag{9}$$

The enthalpies of the specific interactions between the polar solute and the investigated surface are correlated to acidity and basicity:

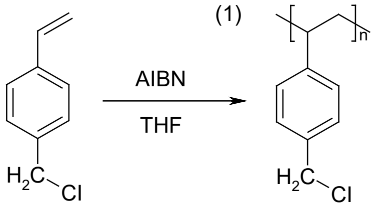
$$-\frac{\Delta H_A^S}{AN^*} = \frac{K_A DN}{AN^*} + K_D \tag{10}$$

where K_A and K_D reflect Lewis acidity and Lewis basicity of a studied solid surface, respectively. A straight line is obtained by plotting $\Delta H_A^S/AN^*$ *vs.* DN/AN^* and the y-intercept and the slope of the line correspond to K_D and K_A value, respectively.

The ratio K_D/K_A provides the experimental characterization of a surface as acidic/basic. Hence, $K_D/K_A > 1$ and $K_D/K_A < 1$ reflect a basic surface and a acidic surface, respectively.

Experimental part

Preparation of poly (vinyl benzyl chloride) (PVBC)



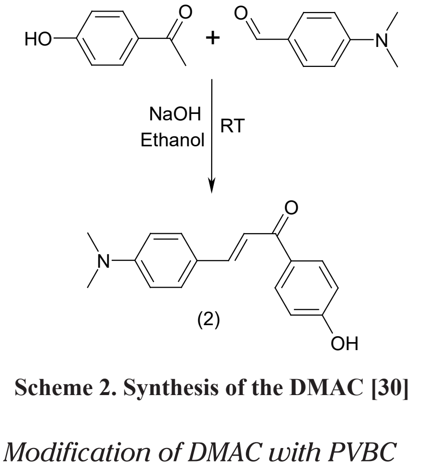
Scheme 1. Polymerization of vinyl benzyl chloride

The 10 mL (0.071 mol) of vinyl benzyl chloride and 0.155 g (0.95 mmol) of AIBN were dissolved in 50 mL of 1-methyl-2-pyrrolidone. Then, the solution was transferred to a 250 mL three-necked flask equipped with a nitrogen inlet, magnetic stirrer, and reflux condenser. The mixture was heated to 70 °C and stirred continuously in a nitrogen atmosphere for five hours (Sheme 1).

The obtained viscous polymer solution was precipitated by using ethanol. The white PVBC (1) was filtered and dried under vacuum at room temperature for 24 hours. Yield was found about as 9.50 g.

Synthesis of 3-(4-(dimethyl amino) phenyl)-1-(4-hydroxyphenyl) prop-2-en-1-one (DMAC)

The 3-(4-(dimethyl amino) phenyl)-1-(4-hydroxyphenyl) prop-2- en-1-one was synthesized by using Claisen-Schmidt reaction. The 5 g of 1-(4-hydroxyphenyl) ethan-1-one (0.0367 mol) was dissolved in 25 mL ethanol and 3.23 g of NaOH (0.0807 mol) in 10 mL of distilled water was added to the solution as a base catalyst. Then 6.6 g of 4-(dimethyl amino) benzaldehyde (0.0444 mol) in 35 mL ethanol was added drop wise to the reaction mixture and reaction was proceeded at 25 °C (RT) for 24 hours (Scheme 2).



Scheme 2. Synthesis of the DMAC [30]

At the end of reaction DMAC (2) was poured in iced water and filtered. Then, the received yellow powder was purified with petroleum ether by Soxhlet extractor. Bright yellow crystals (5.8 g, 0.022 mol) are obtained. (Yield: 60%).

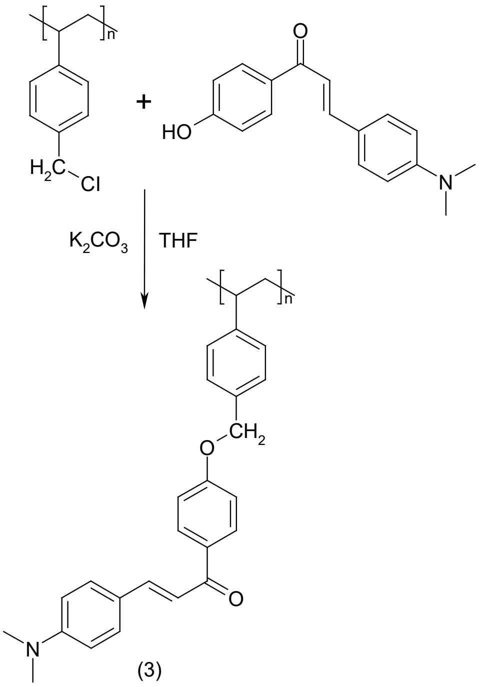
Modification of DMAC with PVBC

The 2 g of PVBC was dissolved in 20 mL of DMF and 2 mL of triethyl amine was added to this solution as an acid scavenger. Then, 3.5 g (0.0131 mol) of DMAC in 10 mL of DMF solution was added to the reaction mixture. The reaction was continued at room temperature for 24 hours and 60 °C for 5 hours. Chalcone modified poly (styrene) based polymer PVBC-DMAC (3) was precipitated in ether (Scheme 3). Obtained polymer was filtered and washed excess of ether and dried under vacuum at room temperature.

The synthesized compounds were characterized using FT-IR spectra. According to the fig. 1., PVBC showed a characteristic C-Cl stretching at about 698 cm^{-1}. The FT-IR spectrum of DMAC shows absorption peaks at 1656 cm^{-1} which is belong to C=O stretching vibration; there is a moderately strong absorption peak at 1585cm^{-1} in spectra shown in fig. 1 which is ascribed to the olefinic, conjugated bond of the chalcone moiety –C=C–stretching vibrations.

On the other hand, C-Cl peak disappear after modification of PVBC with DMAC. Also, in the FT-IR spectrum of chalcone modified PVBC, olefinic absorption band and carbonyl stretching vibrations have been seen at 1594.63 cm^{-1} and 1685.45 cm^{-1} respectively.

An Agilent Technologies 6890N Model gas chromatography with a thermal conductivity detector was used to measure the retention time of the solvents on prepared columns in this study.



Scheme 3. Preparation of chalcone modified poly (styrene) based polymer (PVBC-DMAC)

The used stainless steel tubing (1 m × 3.2 mm o. d.) as IGC column was supplied from Alltech Associates, Inc. PVBC-DMAC was coated on the support material by slow evaporation of DMSO (dimethyl sulfoxide) as stirring the Chromosorb W(AW-DMCS-treated, 80/100 mesh) in the PVBC-DMAC solution. The amount of coated the polymer on the support was determined as 10.68 % by calcination. The 0.1 μL of each solvent was injected into the conditioned column under a helium atmosphere for 24 h at 423.2 K, using a 1 μL Hamilton syringe and flushed into the air.

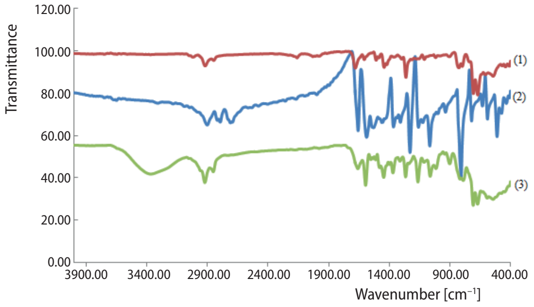


Figure 1. The FT-IR spectra of compounds; PVBC (1), DMAC (2) and PVBC-DMAC (3)

Chromosorb-W (AW-DMCS-treated, 80/100 mesh) as used the support material was received from Merck AG Inc. The silanezed-glass wool used to plug the ends of the IGC column was purchased from Alltech Associates, Inc.

The FT-IR spectra of the synthesized compounds were recorded in a Thermo Scientific Nicolet 380 Spectrometer.

Transition thermal analysis of PVBC-DMAC was carried out using DSC in a Perkin-Elmer calorimeter. For DSC analysis, approximately 14.8 mg of PVBC-DMAC sample in an aluminum pan was heated and cooled at a rate 10 °C min^{-1} from 15 to 135 °C in a nitrogen atmosphere.

Results and discussion

The specific retention volume, V_g^0, values are essential in finding of the glass transition temperature of PVBC-DMAC by IGC method. The V_g^0 is obtained from IGC experiments as follows:

$$V_g^0 = \frac{Q(t_R - t_A)J273.2}{T_f w} \tag{11}$$

where w is weight of PVBC-DMAC in the column [31-35].

The V_g^0 of the acetate and alcohol isomers as solvent nBAc, iBAc, tBAc, nBAl, iBAl, and tAAl on the PVBC-DMAC were determined at temperature range 303.2-353.2 K. The per cent error in V_g^0 was calculated as less than ±0.5 by using four or five successive measurements of each datum. In fig. 2, the retention diagrams are shown a plot of $\ln V_g^0$ obtained from eq. (11) *vs.* 1/T.

As can be seen in fig. 2., T_g value of PVBC-DMAC was found to be 335.2 K from the first point of deviation from linearity towards to higher temperatures in the majority of the plots. Also shown in fig. 2., PVBC-DMAC has capable of seperating for the used isomers in the whole temperature ranges.

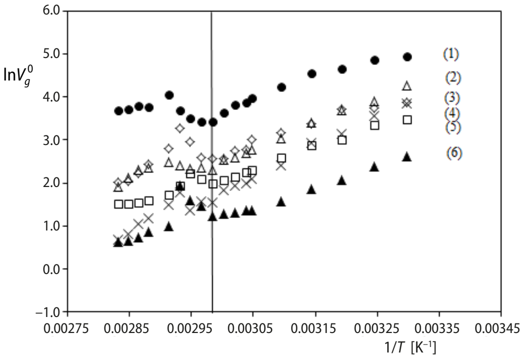


Figure 2. The plot of $\ln V_g^0$ - $1/T$ for the probes; nBAl (1), iBAl (2), nBAc (3), tAAl (4), iBAc (5), and tBAc (6)

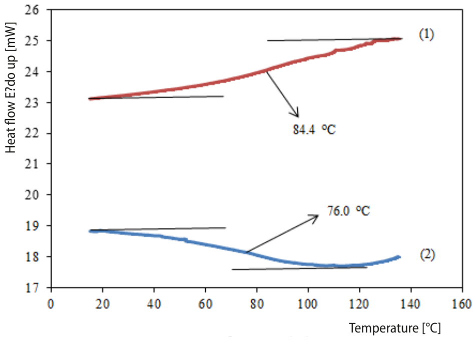


Figure 3. The DSC scans of PVBC-DMAC obtained during the heating (1) and cooling (2) (heating and cooling rates 10 °C/min)

Considering DSC scans of PVBC-DMAC in fig. 3, Tg value was found to be 349.2 K from the cooling curve and to be 357.6 K from the heating curve.

The value of T_g obtained by DSC analysis agree with the one obtained by IGC method. The sorption properties of PVBC-DMAC were examined at infinite dilution by IGC method in the temperature range from 303.2 K to 328.2 K. The net retention volumes V_N, of the polar (DCM, TCM, THF, EA, and Ace) and non-polar (Hx, Hp, O, N, and D) probes on the PVBC-DMAC were calculated using eq. (1). The per cent error in V_N was calculated as less than ±0.5 and each point is average of n = 4 and 5 columns ± standard deviation. In figs. 4 and 5, the $\ln V_N$-$1/T$ graphs for the selected probes are shown.

The dispersive surface energies of PVBC-DMAC as a function of temperature were determined by Dorris-Gray and Schultz methods from using eqs. (3) and (6), respectively. As can be seen in tab. 3, the values of the dispersive surface energies of the polymer were found very close to each other regardless of the method used in the studied experiment temperature region. In this work, we obtained the specific component of the adsorption free energy, ΔG_A^S of the polar probe on PVBC-DMAC as shown by eq. (7), tab. 4.

The plot was given as an example for PVBC-DMAC for 303.2 K in fig. 6. From tab. 4,

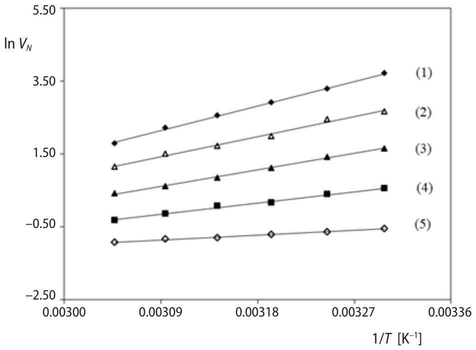


Figure 4. Plot of lnV_n-1/T for the probes; D (1), N (2), O (3), Hp (4), and Hx (5)

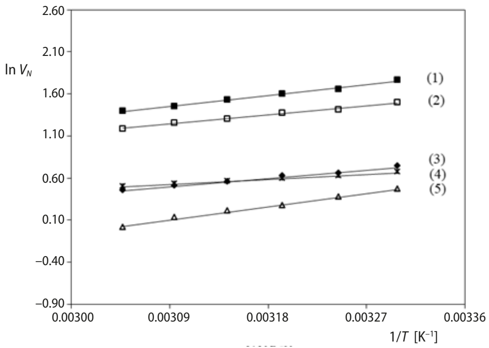


Figure 5. Plot of lnV_n-1/T for the probes DCM (1), Ace (2), TCM (3), EA (4), and THF (5)

Table 3. The values of γ_S^D obtained for PVBC-DMAC

Dorris-Gray Method				Schultz Method	
T [K]	$\gamma_{L[\mathrm{CH_2}]}$ [mJm^{-2}]	$\Delta G_{A[\mathrm{CH_2}]}$ [10^{6}mJmol^{-1}]	γ_S^D [mJm^{-2}]	Slope [·10^{24}]	γ_S^D [mJm^{-2}]
303.2	35.02	2.69	39.45	7.54	39.25
308.2	34.73	2.54	35.62	7.14	35.17
313.2	34.44	2.36	31.05	6.64	30.39
318.2	34.15	2.20	27.24	6.19	26.45
323.2	33.86	2.05	23.83	5.77	22.95
328.2	33.57	1.83	19.08	5.14	18.21

it is clear that the ΔG_A^S values are slightly affected by temperature. The highest ΔG_A^S value was found to be DCM. Then we calculated the specific components of the enthalpies of adsorption ΔH_A^S and the entrophies of adsorption ΔS_A^S of the probes with respect to eq. (8). The calculated values are listed in tab. 5. The values of K_A and K_D were obtained by the graph, fig. 7, drawn according to eq. (10). From the graph, K_A and K_D are found to be 0.0393 and 0.1635, respectively. The obtained value $K_D/K_A > 1$ is confirm that surface of PVBC-DMAC exhibits behavior of Lewis base.

Table 4. The values of ΔG_A^S [kJmol^{-1}] between PVBC-DMAC and the polar probes

T [K]	EA	Ace	DCM	TCM	THF
303.2	3.61	8.74	10.00	2.96	3.11
308.2	3.72	8.60	9.80	3.01	3.07
313.2	3.95	8.66	9.77	3.30	3.13
318.2	4.08	8.53	9.62	3.38	3.14
323.2	4.28	8.58	9.59	3.58	3.20
328.2	4.22	8.45	9.49	3.45	2.89

Table 5. The values of ΔH_A^S and ΔS_A^S of the polar probes on PVBC-DMAC

Solvent	$-\Delta H_A^S$ [kJmol^{-1}]	$\Delta S_A^S \cdot 10^3$ [kJmol^{-1}K^{-1}]
Ace	–11.46	0.84
EA	4.88	5.26
THF	–4.24	1,74
DCM	–15.84	1.24
TCM	4.44	4.71

Conclusion

In this study, we concerned with synthesis and characterisation of PVBC-DMAC. Based on IGC method, we investigated the glass transition temperature and surface properties

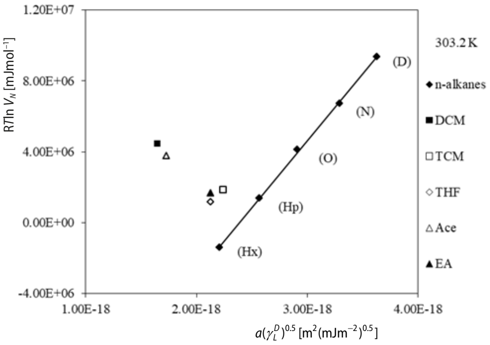


Figure 6. A plot of $RT\ln V_N$-$a(\gamma_L^D)^{0.5}$ for n-alkanes and polar probes on PVBC-DMAC at 303.2 K

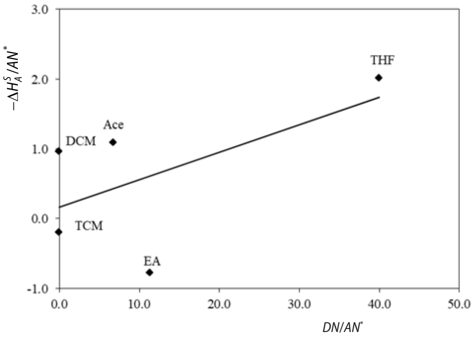


Figure 7. The plot of $-\Delta H_A^S/AN^*$ *vs.* DN/AN^*

of PVBC-DMAC. Then, we concluded that the glass transition temperature of PVBC-DMAC obtained by using IGC measurements is in good agreement with the ones determined by DSC. The results of the study suggest also that the separation ability of the PVBC-DMAC was good enough for the investigated solvents in the studied temperature ranges. The γ_S^D values of PVBC-DMAC have different range from 19.08 to 39.45 mJ/m^2 (Dorris-Gray method) and from 18.21 to 39.25 mJ/m^2 (Schultz method). The values of γ_S^D determined by both calculation method decrease with the increase of temperature in the range of 303.2 to 328.2 K. It was found to be that the surface of PVBC-DMAC is basic.

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Nomenclature[*]

Ace	– acetone	N	– *n*-nonane
D	– *n*-decane	nBAc	– *n*-butyl acetate
DCM	– dichloromethane	nBAl	– *n*-butyl alcohol
EA	– ethyl acetate	O	– *n*-octane
Hp	– *n*-heptane	tAAl	– tert-amyl alcohol
Hx	– *n*-hexane	tBAc	– tert-butyl acetate
iBAc	– iso-butyl acetate	TCM	– chloroform
iBAl	– iso-butyl alcohol	THF	– tetrahydrofurane

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