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Surface characterization of thiazolidinone derivatives by ınverse gas chromatography

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Inverse gas chromatography at infinite dilution was used for the first time to determine the adsorption properties and acid-base contributions to the surface energy of thiazoldin-4-ones bearing heteroaryl substituents. *3-(6-Methyl-2-pyridyl)-2-(2-thienyl)-1,3-thiazolidin-4-one* (MPTT) and *3-(6-Methyl-2-pyridyl)-2-(3-methyl-2-thienyl)-1,3-thiazolidin-4-one* (MPMTT) were freshly synthesized via one-pot three component condensation reaction. The retentions of nonpolar solvents such as n-hexane, n-heptane, n-octane, n-nonane, n-decane and other acidic, basic and amphoteric probes such as tetrahydrofurane, dichloromethane, chloroform, acetone and ethyl acetate used without further purification on MPTT and MPMTT were measured in the temperature ranges from 303 to 333 K by inverse gas chromatography (IGC). The dispersive component of the surface energy , γ_S^D of studied adsorbent surface was estimated using retention times of different nonpolar organics in the infinite dilution region. Dispersive components of the surface energies, γ_S^D according to Fowkes and Dorris-Gray approaches and the acid, K_A and base, K_D constants for the *3-(6-Methyl-2-pyridyl)-2-(2-thienyl)-1,3-thiazolidin-4-one* and *3-(6-Methyl-2-pyridyl)-2-(3-methyl-2-thienyl)-1,3-thiazolidin-4-one* were calculated.

Keywords: Inverse gas chromatography; Thiazolidinone derivatives; Surface and adsorption properties; Surface free energies; Lewis acid-base constants.

INTRODUCTION

Thiazolidinone derivatives have growing interest because of their broad spectrum of activities such as antimicrobial [1], anti-HIV [2], cancer treatment agent [3], essence in cosmetics [4], electron releasing layer in solar cell [5], non linear optical property [6] and so on. Recently reported Quantitative Structure-Activity Relationship (QSAR) [7] and docking studies [8,9] were aimed to understand the pharmacological observations of various thiazolidinones. Docking analysis is good tool to elucidate the potential bindings sites of compounds to the active center of targeted enzyme. Even syntheses and biological activities of thiazolidinones are intensely studied, physical properties of them are known little. Surface characterization and acid-base characteristics of thiazolidinione derivatives will contribute to understanding the binding mechanism.

For this reason, in the present study, the surface free energy and acid-base characteristics of compoundS, MPTT and MPMTT were investigated through measurements of net retention volumes of several probe molecules and by use of adsorption on principle in inverse gas chromatography, IGC. The IGC being simple, rapid, low cost, with good accuracy, and available equipment provides valuable thermodynamic information for the physico-chemical and surface characterization of organic materials such as Schiff bases [10,11], liquid crystal [12].

In this work, the possibility of surface characterization by IGC is illustrated with a study of the sorption properties of MPTT and MPMTT. The stationary phase in IGC was prepared thiazolidinione derivatives covered on chromosorb.

EXPERIMENTAL

Probes and Instrumentation

The used solvents were high purity grade nonpolar solvents such as n-hexane (Hx), n-heptane (Hp), n-octane (O), n-nonane (N), n-Decane (D) and polar solvents such as dichloromethane (DCM, acidic), chloroform (TCM, acidic), acetone (Ac, amphoteric), ethyl acetate (EA, amphoteric) and tetrahydrofurane (THF, basic). The all studied solvents and support materials being Chromosorb-W(AW-DMCS-treated, 80/100 mesh) were supplied from Merck AG. Inc. Silane treated glass wool used to plug the ends of the column was obtained from Alltech Associates, Inc.,

The sorption properties of the MPTT and MPMTT were determined using a Hewlett-Packard 6890 Series II gas chromatograph, with a thermal conductivity detector (TCD). High purity helium was used as the carrier gas (flow rate in the range 25–28 cm^3 min^{-1}). A stainless steel columns (0.5 m

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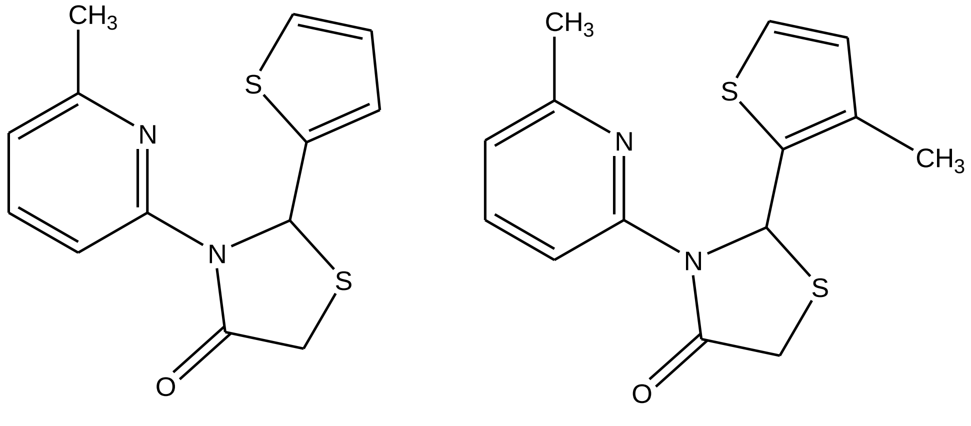

1 **2**

Fig. 1. Chemical Structure of 3-(6-Methyl-2-pyridyl)-2-(2-thienyl)-1,3-thiazolidin-4-one (MPTT) (**1**) and 3-(6-Methyl-2-pyridyl)-2-(3-methyl-2-thienyl)-1,3-thiazolidin-4-one (MPMTT) (**2**)

long, 3.2 mm o.d.) were packed with MPTT and MPMTT covered on chromosorb. After packing, the columns were conditioned overnight at 373 K. Retentions of non polar and polar solvents on MPTT and MPMTT were measured in the temperature range 303–333 K.

Synthesis of 3-(6-Methyl-2-pyridyl)-2-(2-thienyl)-1,3-thiazolidin-4-one (MPTT) and 3-(6-Methyl-2-pyridyl)-2-(3-methyl-2-thienyl)-1,3-thiazolidin-4-one (MPMTT)

3-(6-Methyl-2-pyridyl)-2-(2-thienyl)-1,3-thiazolidin-4-one (**MPTT**) and 3-(6-Methyl-2-pyridyl)-2-(3-methyl-2-thienyl)-1,3-thiazolidin-4-one (**MPMTT**) were prepared according to already known procedure with slight modifications and spectroscopic data are in full agreement with literature [13]. (Figure 1)

INVERSE GAS CHROMATOGRAPHY

The sample under investigation is the stationary phase in inverse gas chromatography while a known substance in the mobile phase acts as a probe molecule. IGC is given information about various adsorption properties, including surface energy, free energy of adsorption and acid–base characteristics. Surface energy is used to describe the energetic situation of a solid surface, being therefore directly related to the adhesion characteristics of a particular material.

In this study, Dorris-Gray and Fowkes methods were applied to determine dispersion component of the surface free energy, γ_S^D of MPTT and MPMTT.

According to Dorris and Gray, corresponding to the adsorption energy of a methylene group, $\Delta G_{A[CH_2]}$ is given by following equation [14]:

$$\Delta G_{A[CH_2]} = -RT\ln(\frac{V_{N,n}}{V_{N,n+1}}) \qquad (1)$$

where $V_{N,n}$ and $V_{N,n+1}$ are the retention volumes of two n-alkanes having n and n+1 carbon atoms in their chain and the adsorbate net retention volumes, V_N were calculated from the expression:

$$V_N = Q * J * (t_R - t_A) * T / (T_f) \qquad (2)$$

where t_R is the adsorbate retention time, t_A is the retention time of air, Q is volumetric flow rate measured at column outlet and at ambient temperature T_f (K), T is the column temperature (K) and J is James-Martin gas compressibility correction factor [15]. The interactions experienced between an adsorbate and an adsorbent can consist of two components-specific and dispersion forces [16]. Dispersion forces, also known as London forces, are present between all molecules, regardless of their identity. Specific forces generally rely on some compatibility between the structures of the interacting molecules, either physically or electronically.

The adsorption energy for the n-alkanes increases with the number of carbon atoms in the chain.This parameter is independent of the chosen state of the adsorbed molecule. Thus at constant temperature, for a series of alkane probes, a plot $RT\ln V_N$ versus the number of carbon atoms should give a straight line from which $\Delta G_{A[CH_2]}$ can be found.

The methylene adsorption energy can also be defined as [14]

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

where N_A is Avagadro's number, $a_{[CH_2]}$ is the surface area covered by one methylene group (0.06 nm^2) and $\gamma_L[CH_2]$ is the surface free energy of a surface consisting of methylene groups, i.e. polyethylene, given by

$$\gamma_L[CH_2] = 35.6 + 0.058(293 - T) \qquad (4)$$

Thus using Eqs.(1)-(4) and the experimentally determined values of $V_{N,n}$ and $V_{N,n+1}$, the dispersion component of the surface free energy, γ_S^D may be calculated according to Dorris-Gray approach.

The retention time of a series of homologous n-alkanes is used to determine the dispersive surface energy of the MPTT and MPMTT from following equation: [17]

$$\Delta G_A^S = RT\ln(V_n) = 2N_A(\gamma_S^D)^{0.5}a(\gamma_L^D)^{0.5} + K'' \qquad (5)$$

where ΔG_A^S is the dispersive free energy of adsorption, γ_S^D is the dispersive componenets of the surface tension of the adsorbent such as MPTT and MPMTT, γ_L^D is the dispersive componenets of the surface tension of the adsorbate such as non-polar and polar solvents. ΔG_A^S is the dispersive free energy of adsorption is calculated from following equation:

$$-\Delta G_A^S = RT\ln\left(\frac{V_{N,n}}{V_{N,ref}}\right) \qquad (6)$$

where $V_{N,n}$ and $V_{N,ref}$ are the retention volume for the polar probe and the retention volume for the n-alkanes' reference line, respectively.

ΔH_A^S is the adsorption enthalpy by Lewis acid-base interactions, ΔS_A^S is the adsorption entropy Lewis acid-base interactions and for each polar probe, ΔH_A^S and ΔS_A^S can be determined from belove equation;

$$\Delta G_A^S = \Delta H_A^S - T\Delta S_A^S \qquad (7)$$

The surface Lewis acidity and basicity constants, K_A and K_D, may be calculated from the equation, [18-20]

$$-\Delta H_A^S = K_A DN + K_D AN^* \qquad (8)$$

The constants K_A and K_D describe the acidity and basicity of MPTT and MPMTT surfaces, respectively.

K_A and K_D are obtained from a plot of $-\Delta H_A^S / AN^*$ versus DN / AN^* with K_A as the slope and K_D as the intercept. Parameters K_A and K_D reflect the ability of the examined surface to act as an electron acceptor and electron donor, respectively [21,22].

Values of $a(\gamma_L^D)^{0.5}$ and boiling point, $T_b(^oC)$ of apolar solvents are found in the literature [21,22]. The values of $a(\gamma_L^D)^{0.5}$ used in this study are presented in Table 1. Values of a, $(\gamma_l^d)^{0.5}$, boiling point, $T_b(^oC)$, the Gutmann's modified acceptor number, AN^* and donor number, DN of the polar probes used in this study are presented in Table 2 [23,24].

Table 1. Values of $a(\gamma_L^D)^{0.5}$ for the selected n-alkane solvents

Probe	$a(x10^{-10}m^2)$	$\gamma_L^D(mJ/m^2)$	$a(\gamma_L^D)^{0.5}(m^2(mJ/m^2)^{0.5})$	$T_b(^oC)$
n-Hexane	51.0	18.4	2.19x10^{-18}	68.7
n-Heptane	57.0	20.3	2.57x10^{-18}	98.4
n-Octane	62.8	21.3	2.90x10^{-18}	125.7
n-Nonane	69.0	22.7	3.28x10^{-18}	150.8
n-Decane	75.0	23.4	3.63x10^{-18}	174.1

Table 2. Values of $a(\gamma_l^d)^{0.5}$, DN and AN^* for the selected polar solvents

Probe	$a(x10^{-10}m^2)$	$\gamma_l^d(mJ/m^2)$	$a(\gamma_l^d)^{0.5}(m^2(mJ/m^2)^{0.5})$	$AN^*(kJ/mol)$	$DN(kJ/mol)$	$T_b(^oC)$
THF	45.0	22.5	$2.13x10^{-18}$	2.1	84.0	66.0
Ac	42.5	16.5	$1.73x10^{-1}$	10.5	71.4	56.0
DCM	31.5	27.6	$1.65x10^{-18}$	16.4	0.0	40.0
TCM	44.0	25.9	$2.24x10^{-18}$	22.7	0.0	61.2
EA	48.0	19.6	$2.13x10^{-18}$	6.3	71.8	77.1

RESULTS AND DISCUSSION

The net retention volumes, V_N of the nonpolar and polar solvents on MPTT and MPMTT compounds were obtained from IGC measurements between 303 and 333 K at infinite dilution region using Eq.(2) and retention diagrams of nonpolar and polar solvents were given in Fig.1a and Fig.1b, respectively for MPTT as an example.

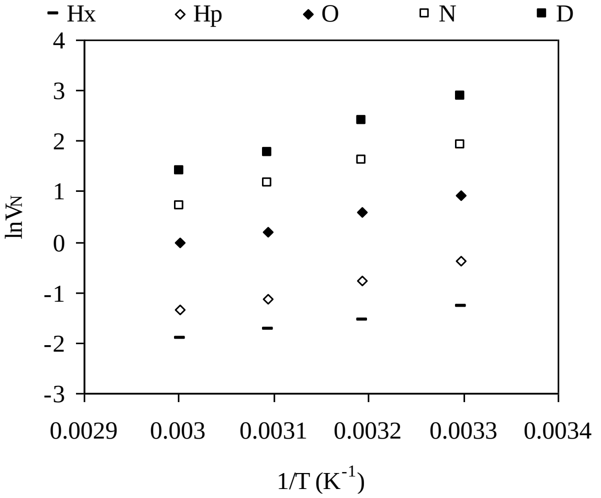

Fig. 1(a). The retention diagram of non-polar solvents adsorbed onto MPTT

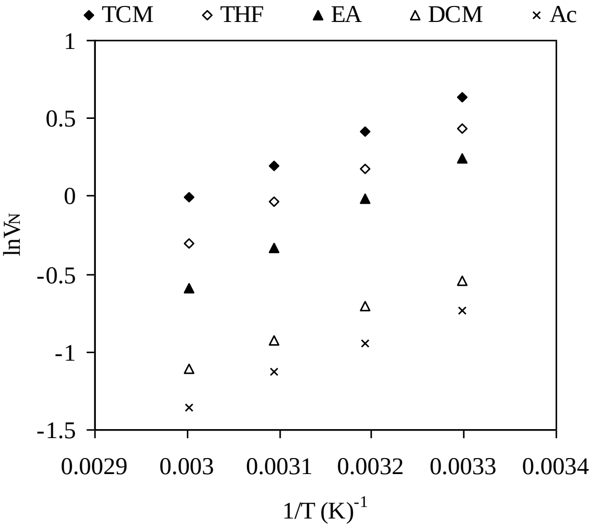

Fig. 1(b). The retention diagram of polar solvents adsorbed onto MPTT

According to Dorris-Gray approaches, Eq.(1), $\Delta G_{A[CH_2]}$ is independent of the chosen reference state of adsorbed molecule. The $RT\ln V_N$ versus carbon number of non-polar solvents were plotted in Fig.(2) for MPTT and Fig.(3) for MPMTT.

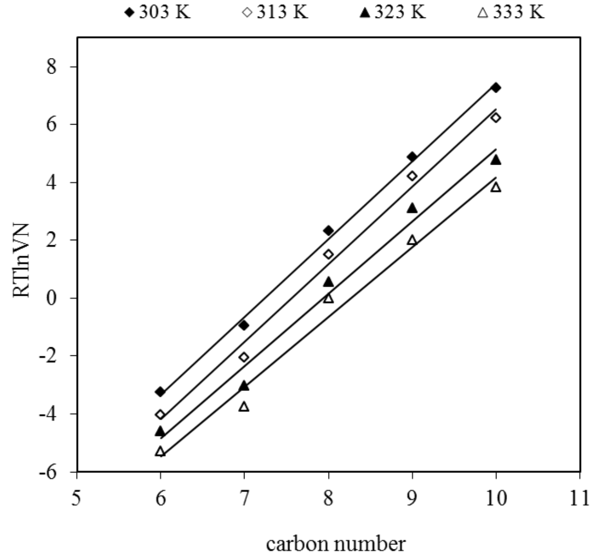

Fig. 2. The plot of $RT\ln V_N$ versus carbon number of non-polar solvents for MPTT

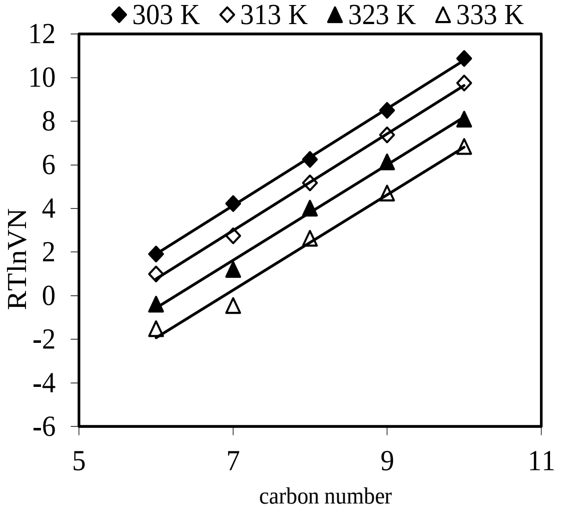

Fig. 3. The plot of versus carbon number of non-polar solvents for MPMTT

The slope of the fitted line is equal to $\Delta G_{A[CH_2]}$. The variation of γ_S^D and $\gamma_L[CH_2]$ with temperature were calculated from Eq.(3) and (4), respectively. The results of $\Delta G_{A[CH_2]}$, $\gamma_L[CH_2]$ and γ_S^D of MPTT and MPMTT were given in Table 3 and Table 4, respectively .

Table 3 The adsorption energy of a methylene group, $\Delta G_{A[CH_2]}$, the surface free energy of a surface consisting of methylene groups, $\gamma_L[CH_2]$ and dispersion component of surface free energy, γ_S^D values calculated by Doris-Gray approach for MPTT determined at studied temperatures

T(K)	$\gamma_L[CH_2]$ (mJ/m^2)	$\Delta G_{A[CH_2]}$ (10^6 mJ/mol)	γ_S^D (mJ/m^2)
303	35.02	2.68	39.45
313	34.44	2.68	39.96
323	33.86	2.49	35.17
333	33.28	2.41	33.33

Table 4 The adsorption energy of a methylene group, $\gamma_L[CH_2]$ the surface free energy of a surface consisting of methylene groups, $\Delta G_{A[CH_2]}$ and dispersion component of surface free energy, γ_S^D values calculated by Doris-Gray approach for MPMTT determined at studied temperatures

T(K)	$\gamma_L[CH_2]$ (mJ/m^2)	$\Delta G_{A[CH_2]}$ (10^6 mJ/mol)	γ_S^D (mJ/m^2)
303	35.02	2.22	27.02
313	34.44	2.21	27.32
323	33.86	2.19	27.12
333	33.28	2.18	27.54

Table 3 and 4 report that the variation of γ_S^D as a function of temperature was limited and the γ_S^D values of MPTT and MPMTT do not change significantly between 303 and 333 K. In comparison, methyl group was decreased the surface energy of thiazolidinone derivative. The methyl group reduced the dispersive surface energy due to the interaction with lower energetic sites in the surface.

The Fowkes equation (Eq. 5, referred to as Schultz and Lavielle approach by some authors) [17] was used to determine the dispersive component of the surface free energy, γ_S^D over a range of studied temperatures and calculated values of $RT\ln V_N$ were plotted against $a(\gamma_L^D)^{0.5}$. An example of the pattern of results obtained was given in Fig. 4 (MPTT) and Fig.5 (MPMTT) for the isotherm at 303 K.

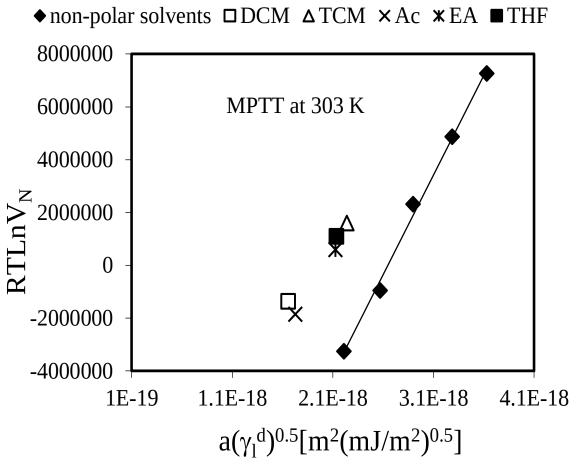

Fig. 4. A plot of $RT\ln V_N$ vs. $a(\gamma_L^D)^{0.5}$ for non-polar and polar probes on MPTT at 303 K

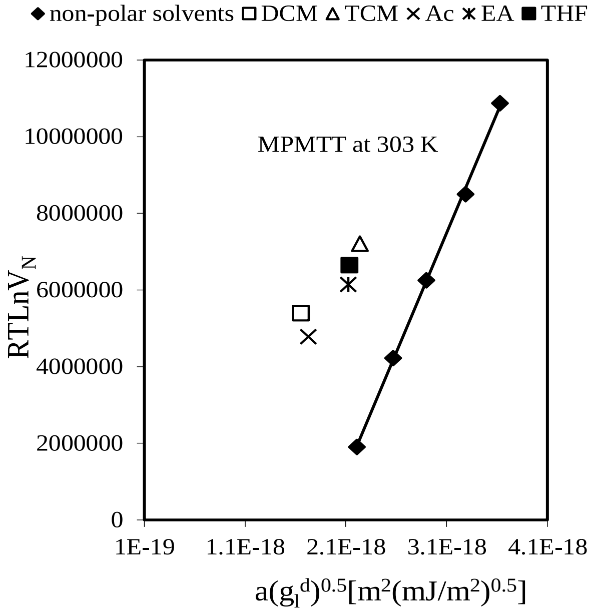

Fig. 5. A plot of $RT\ln V_N$ vs. $a(\gamma_L^D)^{0.5}$ for non-polar and polar probes on MPMTT at 303 K

The linearity was obtained by the non-polar solvents at the studied temperature ranges. The slope ($2N\sqrt{\gamma_S^D}$) of the linear fit, obtained for each n-alkane plot, gives the dispersive component of the surface free energy, γ_S^D at that temperature. The values of γ_S^D at studied temperature ranges were given in Table 5 for MPTT and Table 6 for MPMTT.

Table 5 γ_S^D values calculated by Fowkes approach and slope at studied temperatures for MPTT

T(K)	Slope (x10^{24})	γ_S^D (mJ/m^2)
303	7.5334	39.15
313	7.5195	39.00
323	6.9938	33.74
333	6.7422	31.36

Table 6. γ_S^D values calculated by Fowkes approach and slope at studied temperatures for MPMTT

T(K)	Slope (x10^{24})	γ_S^D (mJ/m^2)
303	6.2370	26.83
313	6.2186	26.68
323	6.1402	26.01
333	6.1348	25.96

The values of γ_S^D of MPTT and MPMTT calculated according to Dorris-Gray and Fowkes approaches are very close each other at the studied temperature ranges. There is no data in the literature to compare these values. Methyl group is decreased the γ_S^D of MPMTT according to MPTT values.

The specific component of the surface free energy, ΔG_A^S , is calculated using the difference between the calculated value of $RT \ln V_N$ and that which was derived using the equation of the linear fit of the n-alkane reference line (Eq.6).

The variation of free energy of specific interactions, ΔG_A^S between MPTT and MPMTT and polar solvents for studied temperatures is given Table 7 and Table 8, respectively.

Table 7. The variation of free energy of specific interactions, $-\Delta G_A^S$ (kJ/mol) between MPTT and polar solvents for studied temperatures

T(K)	*THF*	*Ac*	*DCM*	*TCM*	*EA*
303	*4.9*	*5.1*	*6.1*	*4.7*	*4.5*
313	*5.2*	*5.3*	*6.5*	*5.0*	*4.8*
323	*5.2*	*5.1*	*6.2*	*5.1*	*4.5*
333	*5.1*	*4.9*	*6.1*	*5.2*	*4.4*

Table 8. The variation of free energy of specific interactions, $-\Delta G_A^S$ (kJ/mol) between MPMTT and polar solvents for studied temperatures

T(K)	THF	Ac	DCM	TCM	EA
303	9.1	9.5	10.0	9.5	7.9
313	9.5	9.2	9.7	9.3	7.9
323	9.2	8.9	9.4	9.2	7.6
333	8.9	8.6	9.3	8.9	7.1

The $-\Delta G_A^S$ values of MPMTT were higher than MPTT values. Temperature increasing did not affect significantly the $-\Delta G_A^S$ values of MPTT and MPMTT. ΔH_A^S and ΔS_A^S can be determined from a plot of $-\Delta G_A^S / T$ against $1/T$. (Eq.7)

The values of K_A and K_D were calculated using Eq.(8). The plotting $-\Delta H_A^S / AN^*$ versus DN / AN^* with K_A as the slope and K_D as the intercept. (Fig.6 for MPTT and Fig.7 for MPMTT).

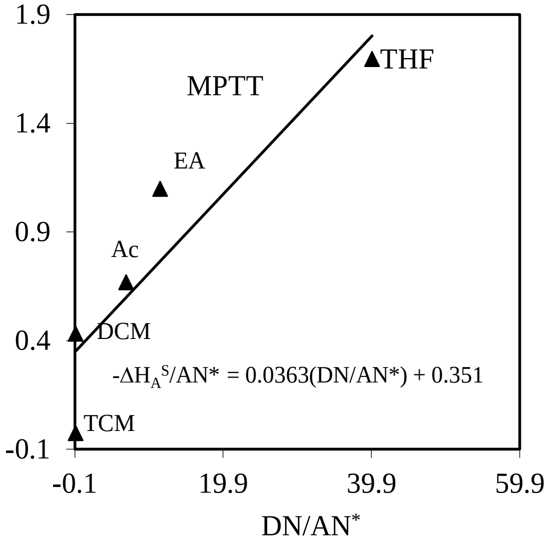

Fig. 6. The plot of $-\Delta H_A^S / AN^*$ versus DN / AN^* for MPTT

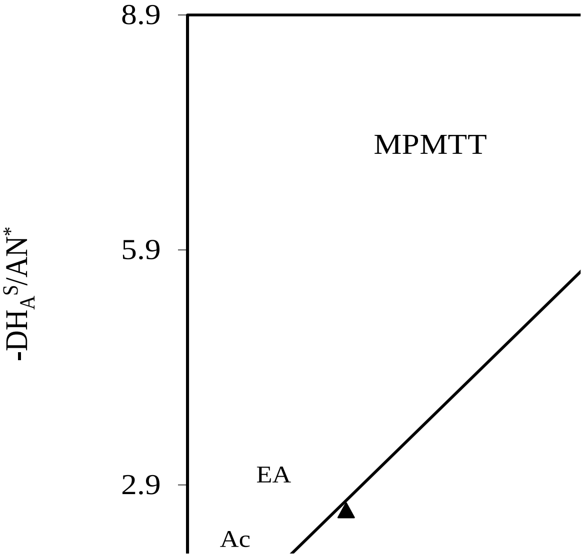

Fig. 7. The plot of $-\Delta H_A^S / AN^*$ versus DN / AN^* for MPMTT

The values of K_A and K_D are found to be 0.04 and 0.4 for MPTT and the values of K_A and

K_D are found to be 0.2 and 0.7 for MPMTT. If the ratio of K_D to K_A is greater than 1, solid surface is basic nature and below 1, it implies that the solid surface is acidic nature [25]. According to the K_D/K_A ratios of MPTT and MPMTT, MPTT surface has more basic nature than MPMTT surface. It can be said that methyl group is decreased the basic nature of thiazolidinone derivatives.

CONCLUSION

Adsorption properties, surface and Lewis acid-base characteristics of MPTT and MPMTT have been examined by means of inverse gas chromatography at infinite dilution, in the temperature range from 303 to 333 K. The γ_S^D values of MPTT change ranges from 39.5 to 33.3 mJ/m^2 and (Dorris-Gray approach) from 39.2 to 31.4 mJ/m^2 (Fowkes approach) with decreasing temperature while the γ_S^D values of MPMTT change ranges from 27.0 to 27.5 mJ/m^2 and (Dorris-Gray approach) from 26.8 to 26.0 mJ/m^2 (Fowkes approach) with decreasing temperature. The values of K_A and K_D parameters indicated that MPTT and MPMTT surfaces are basic nature. IGC is a convenient method for the characterization of the surface and adsorption properties and the acid-base characteristics of thiazolidinone derivatives.

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ПОВЪРХНОСТНО ОХАРАКТЕРИЗИРАНЕ НА ТИАЗОЛИДИНОНОВИ ПРОИЗВОДНИ С ОБРАТНА ГАЗОВА ХРОМАТОГРАФИЯ

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(Резюме)

За пръв път е използвана обратната газова хроматография при безкрайно разреждане за определянето на адсорбционните свойства и киселинните отнасяния за повърхностната енергия на тиазолидин-4-они с хетероарилови заместители. *3-(6-метил-2-пиридил)-2-(2-тиенил)-1,3-тиазолидин-4-он* (MPTT) и *3-(6-метил-2-пиридил-2-(3-метил-2-тиенил)-1,3-тиазолидин-4-он* (MPMTT) са прясно синтезирани чрез едно степенна три-компонентна кондензационна реакция. Измервано е задържането на не-полярни разтворители като n-хексан, n-хептан, n-октан, n-нонан, n-декан и други киселинни, базични и амфотерни проби, като тетрахидрофуран, дихлорметан, хлороформ, ацетон и етилацетат без пречистване на MPTT на MPMTT в температурния интервал от 303 до 333 K чрез обратна газова хроматография (IGC). Дисперсионната компонента на повърхностната енергия γ_S^D на изследваните адсорбционни повърхности е оценена използвайки времената на задържане на различни неполярни органични съединения в областта на безкрайно разреждане. Дисперсионните компоненти на повърхностните енергии γ_S^D по Fowkes и Dorris-Gray са близки и са пресметнати киселинните K_A and базичните константи K_D за съединенията *3-(6-метил-2-пиридил)-2-(2-тиенил)-1,3-тиазолидин-4-он* и *3-(6-метил-2-пиридил-2-(3-метил-2-тиенил)-1,3-тиазолидин-4-он.*